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July 1934

A REVIEW OF THE PATENTS AND
LITERATURE ON THE MANUFACTURE
OF POTASSIUM NITRATE WITH NOTES
ON ITS OCCURRENCE AND USES

By

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and

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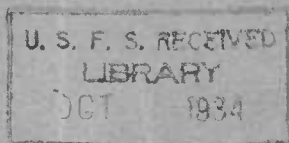
Division of Fertilizer Technology, Fertilizer Investigations

Bureau of Chemistry and Soils



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By COLIN W. WHITTAKER, *associate chemist*, and FRANK O. LUNDSTROM, *assistant chemist*, Division of Fertilizer Technology, Fertilizer Investigations, Bureau of Chemistry and Soils

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INTRODUCTION

Potassium nitrate is one of the oldest known salts. Although the ancients were interested principally in its use in fireworks and explosives, it is probably also one of the oldest artificial fertilizers. Interest in this salt is keen today because of the trend in fertilizer practice toward the use of concentrated fertilizers. As is pointed out later,

potassium nitrate is particularly well adapted for use either as a direct fertilizer or for admixture with other plant-food materials in the manufacture of complete fertilizers. Its high cost, as compared with that of other sources of nitrogen and potash, has so far sharply limited its use in fertilizer practice. The development of a process that will render this salt inexpensive enough for general fertilizer use is a consummation much to be desired.

The recent large decrease in the cost of ammonia, owing to the development of synthetic processes, has suggested the possibility of producing saltpeter cheap enough for fertilizer purposes from potassium chloride and nitric acid, or oxides of nitrogen, both of which are prepared by the oxidation of ammonia. Although a large part of the activity centers in this field, developments along other lines have not been lacking. Large numbers of patents have appeared and continue to appear on a wide variety of processes including double decomposition, nitric acid digestion of various potassium salts or potash silicates, and various combination procedures.

In fact, the whole aspect of the potassium nitrate problem has changed greatly in recent years chiefly because of the two factors mentioned, the trend toward concentrated fertilizers, and the decrease in the cost of nitrate nitrogen, owing to the development of processes for producing cheap ammonia.

Most of the potassium nitrate on the market today is produced by the conversion process from sodium nitrate and potassium chloride (p. 15). Processes in which sodium nitrate is replaced by ammonium, magnesium, or calcium nitrates, or by nitric acid, and the potassium chloride by crude salts, such as sylvine or kainite, containing it, are becoming increasingly important in Europe, especially in producing potassium nitrate for agricultural purposes. Frequently these processes are made to yield a mixed fertilizer which contains potassium nitrate or its ions, without the actual separation of that salt as such. No data are available to show what part of the present world production is due to these newer processes.

Potassium nitrate, in addition to its use as a fertilizer, is employed in the curing of meats, in the manufacture of match-head and pyrotechnic compositions, dynamites, black powder, glass, ceramic glazes, and candles. This salt finds use also in the pickling of candlewicks; processing of tobacco; as a diuretic, diaphoretic, and antiseptic in medicine; as a laboratory reagent; to some extent in metallurgy and enameling. It has other minor uses as well.

In this publication the history and occurrence of potassium nitrate are discussed briefly, and the various processes in actual or prospective use for its preparation are described.

A large part of the literature used in developing the main body of this publication consists of patents. The statements of the inventor are taken for the most part at face value with no attempt at critical comment. It is not possible to state here under what patents the processes now in use are operating, and where patent references are given in connection with such processes they are intended merely as examples of patents obtained in that general field.

Patents that describe or have a bearing on a given process are listed alphabetically under that process in the section entitled "Patent Literature." It has not been possible to discuss or even mention all these patents in the text. Those that are referred to in the text will

be found under the same heading in Patent Literature, together with other patents of interest in connection with the particular process.

The patents have here been classified primarily according to the type of reaction which was employed in that step of the process in which the potassium nitrate was produced. An invention according to which calcium nitrate was first prepared by some special means and then used to carry out a double decomposition with potassium sulphate has therefore been cited under double decompositions with potassium sulphate and calcium nitrate. Many patents could legitimately have been placed under two or more headings, but this has not been done, except in the more important cases. It was difficult at times to decide whether or not a patent should be cited. In general, all patents are included in which potassium nitrate or alkali nitrates are prepared. A few patents which claim only sodium nitrate are included because such processes could in some cases be used for potassium nitrate also. Patents restricted to the production of ammonium nitrate, alkaline earth nitrates, or heavy metal nitrates are not mentioned. For the sake of historical background, several very old processes are described.

HISTORICAL SKETCH

Potassium nitrate (saltpeter) was probably first used in the preparation of fireworks or gunpowder. It is supposed to have been a component of the Greek fire invented in the latter part of the seventh century. The first definite reference to potassium nitrate, or saltpeter in the literature was made in connection with gunpowder. Europe probably learned of gunpowder from the Saracens in the twelfth or thirteenth century. Roger Bacon and Albert Magnus both refer to this salt in the thirteenth century. The history of saltpeter is, indeed, inextricably tied up with the history of fireworks and gunpowder. The early chemists are supposed to have been unable to distinguish between sodium and potassium nitrates, but they must have recognized that some nitrates were more suitable for gunpowder than others. Sodium nitrate is not well adapted for this purpose, owing to its hygroscopicity. Mellor (46, p. 802)¹ states that J. Bohn first clearly distinguished between the crystals of potassium and sodium nitrates about 1683. This difference was emphasized by J. G. Wallerius in 1750, by J. B. L. Rome de l'Isle in 1783, and by R. J. Haüy in 1801.

Potassium nitrate has been called by many names. The confusion of sodium with potassium nitrates in earlier times and, indeed, the confusion of all salts with each other makes the early terminology difficult to trace. The aqueous extract of vegetable ashes was called neter by the ancient Hebrews. This substance was referred to by Jeremiah (2:22) in the seventh century, B.C., and neter was translated as nitre. The neter (nitre) of Solomon (Proverbs 25:20) was apparently sodium carbonate, since it effervesced when vinegar was poured on it. The nitrum of the Romans and nitron of the Greeks refer to the same thing as the neter (translated nitre) of the Hebrews. The lixivium of wood ashes, the impure sodium carbonate from trona deposits and saline lakes of Egypt, and the saline efflorescence on walls—sal murale—all seem to have been called nitrum by the ancients.

¹ Italic numbers in parentheses refer to Literature Cited, p. 50.

The alkaline efflorescence on soils in hot countries was also known by this name. It is evident, therefore, that potassium and sodium carbonates and nitrates were thus confused under one term. It is not known when potassium nitrate was first recognized as something distinct. About the twelfth or thirteenth century the term "sal petrae" (salt of the rock) was applied to that form of nitrum which could be used as one of the main ingredients of gunpowder or Greek fire. The term "sal nitri" is applied to this salt in the writings of the later alchemists. The term "saltpeter" is used extensively in the industry today. Niter is still used to some extent but is sometimes applied also to sodium nitrate.

The terms "sal nitrum", "sal petrae", and "sal prunelle", the latter referring especially to the white fibrous mass obtained by fusing potassium nitrate and allowing it to cool, are still sometimes used in pharmacy, although the more modern terms "niter", "saltpeter", or "potassii nitras" are in more general use. In the chemical industry the term "saltpeter" is used quite generally. Sodium nitrate is usually known as Chile saltpeter, whereas calcium nitrate is sometimes called wall saltpeter, or Norwegian saltpeter. Caution must be exercised in translating the German word "Salpeter." Although its real meaning is potassium nitrate, in common usage it is generally applied to the sodium compound (32, p. 653).

The history of saltpeter has been dealt with at length by several writers (4, 5, 6, 7, 11, 34, 35, 36, 38, 39, 41a, 42, 56, 64). These references include also some of the older literature on processes not cited elsewhere.

STATISTICS OF THE SALTPETER INDUSTRY

The potassium nitrate, or saltpeter, industry has changed little in recent years. It is difficult to present an accurate picture of the industry from such production and consumption figures as are obtainable, chiefly because production of saltpeter has never been a major industry and for that reason has received comparatively little attention from the various agencies that compile industrial statistics.

Of 29 countries for which exports and imports of saltpeter are reported for 1929 (30), exports exceeded imports in four, namely, in Germany, France, Netherlands, and British India. Table 1 shows the imports and exports of these 29 countries in 1913 and from 1926 to 1931, inclusive. No figures for Chile (compare table 2) were given by this source.

Since there are no known deposits of crude potassium nitrate of commercial value in the United States, domestic consumption is dependent on the manufacture or importation of this salt. Production statistics for refined potassium nitrate are not reported by the Bureau of the Census. It is believed that no large amount of potassium nitrate, either crude or refined, has been manufactured or produced in the United States in recent years. No production was reported to the Bureau of the Census in either 1929 or 1931.

At present the principal sources of supply of potassium nitrate for the United States are Germany and Chile. Three grades are being received from Germany, 95 percent, 98 percent, and 99.8 percent. The 99.8 percent grade is termed "double refined", in the trade, and it is used principally in the gunpowder-making and meat-packing industries. It carries a duty of 1 cent a pound. The other two

grades are imported largely for agricultural purposes and are not dutiable. The product received from Chile, sometimes called Chilean potassium nitrate (p. 13) or simply potash nitrate should not be called potassium nitrate, as the content of that salt is less than 50 percent. It is, however, being used rather extensively in the fertilizer industry and must therefore be included in any discussion of sources of potassium nitrate. This product is also known as high-potash nitrate and as nitrapo. It contains from 25 to 40 percent of potassium nitrate. The average of 63 shipments over a period of 3 years was 29.27 percent of potassium nitrate.²

TABLE 1.—Imports and exports (metric tons) of saltpeter by countries ¹ ², 1913 and 1926–31

IMPORTS

Country	1913	1926	1927	1928	1929	1930	1931
Germany ³	402	943	213	342	155	67	12
Belgium.....	85	7	37	134	25	53
Estonia.....	21	24	13	73	16	21
Irish Free State.....	603	218	199	151	168
Finland.....	36	42	64	38	36	51
France ³	71	22	14	235	281	213	182
Great Britain and Northern Ireland ³	12,085	6,224	6,715	6,214	6,345	5,397	4,537
Greece ³	519	75	79	113	160	158	134
Italy.....	1,530	1,615	1,868	1,799	147	2,536
Lithuania.....	26	33	54	26	36	93
China.....	1,747	1,492	1,834	1,501	1,378	1,678
Indo-China.....	110	159	99	152	140	163	60
Japan.....	421	404	209	882
Algeria.....	82	28	26	45	102	100
Mauritius.....	2,083	2,402	3,295	2,832	3,277	4,997	2,524
Tunis.....	25	10	36	40	46	120	6
Union of South Africa.....	114	97	108	113	113	111
Australia.....	571	660	459	714	1,058	488
Norway.....	522	125	124	140	150	156	119
Netherlands ³	424	697	875	980	1,527	1,458
Poland.....	388	558	288	498	418	481
Portugal.....	479	239	245	320	409	370	233
Rumania.....	177	77	722	123	151	54
Czechoslovakia.....	300	546	425	586	502	546	551
Yugoslavia.....	1	18	76	5,246	53	47
Canada.....	14	532	126	38	28	27
Argentina.....	747	598	455	642	429	950
.....	164	82	151	113	75	174	148

EXPORTS

Germany ³	16,058	13,107	21,244	23,161	28,535	26,000	36,247
Belgium.....	285	639	142	67	108	14
Irish Free State.....	1	0
France ³	708	8,361	7,616	6,899	6,746	4,167	3,591
Great Britain and Northern Ireland ³	1,723	891	888	841	660	589	729
Italy.....	2	1	2	1	1	3
British India.....	14,694	5,021	6,250	4,550	4,659	3,888	6,255
Netherlands ³	552	2,145	1,836	1,226	992	300
Poland.....	71	41	6	62	0	1
Rumania.....	2	0	0	0	0	0	0
Sweden.....	1	0	1	3	1	2	4
Czechoslovakia.....	98	99	107	97	0
Yugoslavia.....	0	0

¹ Annuaire International de Statistique Agricole (1929–30) Rome 1930. (50) Abridged from table 311, trade statistics of potash fertilizers.

² Quantities intended for agriculture not specified.

³ The data for 1913 are not comparable with those for 1926 to 1929.

The Tariff Act of 1846 specified a duty of 5 percent on crude saltpeter imports and 10 percent on both the refined and partly refined product. These duties were reduced somewhat in 1857, and in 1861 a duty of 1 cent a pound was imposed on crude saltpeter and 2 cents a

¹ Information obtained by correspondence with W. R. Grace Co., New York City.

pound on both partly refined and refined saltpeter. The duty on partly refined saltpeter remained at 2 cents a pound until 1883, after which date only the two grades, crude and refined, are listed in the various tariff acts. The duty on crude saltpeter rose to 2½ cents a pound in 1864 but fell to 1 cent a pound in 1872, where it remained until 1890, when it was put on the free list, and it is still on that list. The duty on the refined product rose to 3 cents a pound in 1862 and continued until 1872, when it was reduced to 2 cents. In 1883 it fell to 1½ cents, to 1 cent in 1890, and to one-half cent in 1894, where it remained until 1913, when it fell to 0.312 cent (expressed in the Tariff Act as \$7 per ton). The Tariff Act of 1922 returned the duty to one-half cent, but in 1930 it went back to 1 cent for the first time since 1890.

Prices paid for crude potassium nitrate have varied considerably with the source, owing probably to differences in purity. In order to follow the price trend, the yearly values of imports from India given in Foreign Commerce and Navigation of the United States (59) were calculated to price per pound. Subsequent to 1920, when India ceased to export important quantities to the United States, the figures are based on the German product. Crude saltpeter was selling for 3.61 cents a pound in 1850, but it rose to about 6 cents in 1858 and continued at that price until 1866, when it fell to about 3.5 cents. By 1868 it had fallen to 3.1 cents but rose in price the following year and reached 4.76 cents in 1873. From this level it declined steadily but slowly to 2.66 cents in 1888. From 1888 to 1915 it fluctuated between the extreme limits of 2.10 cents and 3.66 cents but was generally between 2.5 cents and 3 cents. In 1916 it fell off sharply to 1.38 cents. The year 1917 saw an extreme peak of 18.27 cents; 1918, a decline to about 13 cents; and by 1920, the price was back to 5.7 cents (Germany). In 1923 saltpeter sold for 2 cents a pound, but since that time the price has varied between 2.4 and 4 cents, the 1929 value being 3.7 cents, which fell to 2.5 cents in 1932.

General imports of crude potassium nitrate into the United States by countries every tenth year from 1850 to 1900, inclusive, are given in table 2, the imports in the years 1905, 1910, and 1913 are then given, and thereafter the imports for each year are shown. Since 1850 the United States has imported crude saltpeter from a total of about 50 countries and localities including Peru, Chile, Egypt, Portuguese Africa, India, China, Russia, Germany, France, Mexico, and Canada. Between 1850 and 1910, however, most of our crude saltpeter was imported from British India. In 1910 Canada exported 13,000,000 pounds to the United States, as compared with 11,000,000 pounds from India. The original source of this Canadian saltpeter is not known to the writers. In other years of the period covered, imports from Canada were insignificant. Exports from India to the United States continued large in volume through 1920, when this country imported about 9,700,000 pounds. The following year the imports fell to less than half that figure and by 1922 were less than 1,000,000 pounds. Since that time imports from India have been negligible. Imports from Germany first became important in 1900, when 670,000 pounds were exported to the United States. The amount varied sharply up to the time of the World War, during which period none was received from that source. A small amount was received in 1920. Since that time imports from Germany have increased irregularly.

TABLE 2.—General imports of crude saltpeter from various countries into the United States in stated years ^{1 2 3}

Country	1850	1860	1870	1880	1890 ⁴	1900	1910	1913	1914	1915	1916	1917	1918	1918 ⁵
England	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons
British India	6,769	7,537	4,765	5,863	4,313	5,390	5,047	2,753	1,533	300	1,912	2,278	3,087	860
Canada							5,936	10			494	204	115	2,602
British possessions, except Canada and British India											54			
Germany		659		116									634	634
Japan			725	31	300	505	201	2,216	50					
China, including Hong Kong and Singapore											10	5	25	
Cuba			1											
Chile ⁶	218	841								3				
Peru	31	565										1,548	14	
Belgium						17								
Mexico						6						60	62	
Norway								25						75
Other countries														
Total	7,018	9,002	4,836	6,319	4,795	4,613	5,919	11,184	5,005	303	2,470	4,095	3,937	4,171

Country	1919	1920	1921	1922	1923	1924	1925	1926	1927	1928	1929	1930	1931	1932
England	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons	Long tons
British India	523	243												
Canada	2,712	4,316	1,766	317	25	75	1,999	1,608	123	397	773	2,856	6,690	12,119
Germany			199						50	98	2	88	400	410
Netherlands		51		15	637	164			89	2				31
Cuba						1								
Chile ⁶	7													
Belgium	13,236	12,156	5,443	2,069	2,047	430	6,251	7,765	3,982	8,910	11,849	9,371	8,270	4,315
Mexico					18		146	19		73				
Norway	30	34												
France					20	19								
Other countries	300													
Total	16,998	16,899	7,493	2,435	2,747	689	8,396	9,416	4,309	9,674	12,800	12,799	15,509	17,067

¹ The fiscal year 1850 to 1918 was from July 1 to June 30, e.g., the fiscal year 1916 started July 1, 1915, and ended June 30, 1916. Beginning in 1918 the fiscal year coincides with the calendar year.

² Preliminary figures show 25,593 long tons from all sources in 1933.

³ Compiled from Foreign Commerce and Navigation of the United States (59) by James H. Shimp.

⁴ Duty free subsequent to Oct. 6, 1890.

⁵ Calendar year from January to December.

⁶ Hamburg.

⁷ Bremen.

⁸ High-potash nitrate. See p. 13.

⁹ Argentina, 235 tons; United Kingdom of Great Britain, 1.

¹⁰ United Kingdom of Great Britain.

Total imports of refined potassium nitrate entered for consumption (59) (not shown in table 2) in the years 1923 to 1932, inclusive, ranged between 420 long tons (1930) and 8,068 tons (1929). Over 2,000 tons were imported in all but two of these years, 1930 and 1932. In most years between 1900 and 1923, 100 to 200 tons were received but over 1,000 tons were imported in 1910.

Chile began to export important quantities of high-potash nitrate to the United States (3,500,000 pounds) in 1917. In the absence of complete data on the potassium nitrate content of this high-potash nitrate, it is difficult to state whether Germany or Chile is our most important source. Germany supplies nearly all that used for other than agricultural purposes. Potassium nitrate contained in mixed fertilizers imported into the United States is not included in table 2.

POTASSIUM NITRATE AS A PLANT FOOD

The three most important fertilizer elements are nitrogen, phosphorus, and potassium. Broadly speaking, fertilizers are priced on the basis of their content of these three elements which are expressed, respectively, as nitrogen (N) or ammonia (NH_3), phosphoric acid (P_2O_5), and potash (K_2O). Ordinarily in the manufacture of artificial fertilizers it is necessary to mix together various materials, each of which usually contains only one of the above elements, in order to obtain a fertilizer that contains all the important fertilizing elements. Saltpeter, however, contains two of the essential fertilizing elements, associated in the same molecule, namely potassium and nitrogen, and thus is a better source for these two elements, so far as quantity or bulk is concerned, than the materials ordinarily used. The physical properties of this salt also make it particularly adapted both for direct use as a fertilizer and for mixing with other materials to produce complete fertilizers. Mehring (44) has shown that the drillability of a fertilizer, that is, the ease with which uniform distribution is obtained with a fertilizer drill, is determined largely by its moisture content. The moisture content of a fertilizer after being shipped or stored for short or long intervals depends on its hygroscopicity or the ease with which it takes up moisture. Adams and Merz (1) found potassium nitrate to be among the least hygroscopic of all fertilizer materials. Potassium nitrate has little or no conditioning effect in itself when mixed with other fertilizer materials, but the quantity of conditioner required to maintain it in good mechanical condition is much less than for any other nitrate. Its use is therefore very desirable in concentrated mixtures which require nitrate nitrogen as an essential component. Potassium nitrate also has the advantage of containing two fertilizer elements in the same molecule. This avoids the trouble caused by segregation which often occurs during the shipping and handling of mixtures.

Although the potash (K_2O) content of potassium nitrate is lower than that of other common sources of potash (931.6 pounds K_2O to 1 short ton of nitrate, 1,081 pounds a ton of sulphate, and 1,263.4 pounds a ton of chloride), its nitrogen content brings its plant-food content up to 1,158.7 pounds a ton, which is higher than that of the sulphate and slightly lower than that of the chloride. The chlorine content of the chloride or muriate is harmful to some crops.

Potassium nitrate has the advantage, as have anhydrous ammonia and urea, of being a fertilizer material that does not impart any non-fertilizing inorganic residue to the fertilizer or to the soil.

Urea and ammonium salts, especially ammonium sulphate, tend to produce an acid soil reaction, but nitrogen may be applied as potassium, sodium, or calcium nitrate without bringing about this condition. Although certain crops, such as rice and other irrigated plants, prefer ammonia nitrogen, a higher rating is usually given to nitrate nitrogen for use on most other crops.

Potassium nitrate and sodium nitrate behave similarly in the soil, with respect to their nitrogen content. The sodium and potassium, however, show considerable differences in their behavior. No great amount of information is available regarding the disposition of sodium, but the indications are that it remains either water soluble or in a form readily exchangeable by other bases, and it is leached out with comparative ease. The behavior of water-soluble potassium in soils has been more extensively studied. Hoagland and Martin (26) found that water-soluble potassium behaves very differently when added to different soils. In some soils it all remains in exchangeable and readily available form. In other soils much of it is fixed in a non-replaceable form, too insoluble to be readily available for plant growth. In any event the loss of potassium from soils by leaching is comparatively small.

The advantages discussed are of no importance unless the fertilizing action of potassium nitrate compares favorably with that of other materials commonly supplying nitrogen and potash. Theoretically, it was not to be expected that the nitrogen or the potash of potassium nitrate would act differently than that from other inorganic sources, and experiments have confirmed this expectation.

Frowein (19), as a result of experiments on six standard crops at two German experiment stations, found that in general potassium nitrate gave results at least equal to equivalent amounts of potash and nitrogen from other materials. Wheeler and Tillinghast (61) concluded from the results of 5-years' experimentation on grass and beets that there was no striking difference between yields obtained with potassium nitrate and those obtained with like quantities of nitrogen and potash, as sodium nitrate and potassium chloride. Edler (15) found potassium nitrate superior to sodium nitrate for fertilizing potatoes, and Maynard (43) obtained varying results on carnations. In experiments on tea culture, Wiles (63) found that potassium nitrate continued to show a marked beneficial effect 5 years after the application.

Other workers (8, 25, 40, 47, 53, 60) have investigated the action of potassium nitrate on a variety of crops grown under various conditions. The results of these and other experiments have definitely established the worth of potassium nitrate as a source of both nitrogen and potash.

OCCURRENCE OF POTASSIUM NITRATE

The potassium of naturally occurring saltpeter is probably derived through the weathering of feldspathic rocks. The nitrogen is supplied from the atmosphere or from decaying organic matter, the nitrates commonly found in soils being formed from the latter. In moist climates these salts are dissolved in the ground water and are washed

away or consumed by growing plants. In arid or protected regions, nitrates, especially sodium nitrate, may accumulate to considerable extent. Small pockets containing almost pure saltpeter occur in widely scattered areas, but no large deposit containing a high percentage of potassium nitrate has ever been found. The sodium nitrate beds of Chile perhaps contain the world's largest supply of potassium nitrate, but the concentration, although varying widely, is generally low. According to Mellor (46, p. 803), caliche, the crude nitrate ore, contains from 0.3 to 26.1 percent of potassium nitrate.

Bryant (10, p. 361*T*) has given an account of the extensive potassium nitrate deposits that occur in the northwestern part of Cape Colony and in the Transvaal. He states:

The nitrate deposits occur in the Griquatown beds of the Transvaal geological system; they extend from just south of the Orange River northward to the Kalahari Desert, and then eastward into the Transvaal from Zeerust to Pietersburg * * *

The nitrate deposits occur in the middle portion of these beds, in the softer and more decomposed shales. Occasional large lumps of salt weighing from 5 to 10 pounds each and frequently analyzing more than 50 percent potassium nitrate fill the cracks or occur in pockets at the bases of strata that have been cut through by streams or otherwise. The nitrate shales show a soluble content ranging from 3 to 10 percent, of which the main constituent is everywhere potassium nitrate. These shale beds have an area of some hundreds of square miles, and their average thickness is 10 feet. The nitrate content is at least 3 percent of the weight of the shales.

Small deposits of saltpeter in this region have also been attributed to the habit of rock rabbits depositing their excrement in particular spots. This substance is rich in nitrogen, and if the rocks on which it is deposited contain potash, as many rocks do, it is conceivable that potassium nitrate would be formed.

In spite of the apparent large reserves of potassium nitrate available in South Africa, it appears that they are used only for supplying local needs. One such deposit supplied the old Boer Government with saltpeter for making gunpowder.

Potassium nitrate is found in the soil in many places but particularly around villages in India, Mexico, Egypt, Persia, Hungary, Italy, China, and elsewhere. Its presence there is ascribed to the high organic content of such soils, owing to inadequate disposal of excrement, combined with the presence of potash-bearing rocks or wood ashes. This source of nitrate is further discussed later.

Mexico was thoroughly searched for saltpeter deposits during the World War. In the States of Guanajuato, Queretaro, Michoacan, and San Luis Potosi, pockets were discovered containing approximately 20 tons of earth and running as high as 60 percent nitrates, most of which was potassium nitrate. These deposits have been worked by the natives by simple leaching with hot water and treatment of the lixivium with ashes of the agave plant. The resulting solution was evaporated and allowed to crystallize. Deposits of volcanic ash, characterized as extensive and containing from 1.5 to 6.5 percent of saltpeter (including calcium and ammonium nitrates) are found in Mexico. Probably none of these deposits is of more than local importance (3).

Saltpeter is reported in other countries in small pockets or in deposits of low concentration. It impregnates thick dolomitic lime-

stone beds near the village of Tzesi, Caucasus. Analysis of the rock gave 0.80 percent KNO_3 (55). The saltpeter deposits of China have been described by Brücher (9), and Read (51) has discussed those of inner Mongolia. A deposit occurring in a shallow cavern near Goyder's Pass, MacDonnell Range, Central Australia, has been reported by Mawson (41).

The nitrate deposits of the United States have been described by Gale (20). Saltpeter deposits are reported in North Carolina, Alabama, Arkansas, Illinois, Indiana, Kentucky, Missouri, Tennessee, West Virginia, Wyoming, Colorado, Idaho, Utah, Oregon, Nevada, California, Arizona, Texas, and New Mexico (18). They consist usually of cave deposits (37, 41a); that is, deposits occurring in protected spots where conditions are favorable. As a rule, these deposits are of very small extent and offer no prospect of ever supplying our demand for saltpeter. The nitrate deposits of southeastern California have been exhaustively investigated (48).

It does not appear that saltpeter, in spite of its wide occurrence on the face of the earth, will ever be other than a manufactured product. Small deposits of cave niter have played their part in past emergencies and may do so again in the future.

PRODUCTION OF POTASSIUM NITRATE

SALTPETER FROM THE SOIL

As noted elsewhere, saltpeter is a soil constituent in many localities, but in only a few places is the concentration high enough to make recovery feasible or profitable. Saltpeter production from the soil is or has been practiced in India, China, Mexico, Guatemala, Egypt, Turkey, Tibet, Burma, Turkestan, Ceylon, Hungary, Poland, Spain, Brazil, and Peru. In the more temperate countries, it has been produced in times of emergency from the earths of caves, cellars, and other protected places. The production of saltpeter from Kentucky caves has been described by Maxson (41a). The saltpeter industry of India is very old, and up to about 1860 India was practically the only source of this material.

IN EAST INDIA

In manufacturing saltpeter, the East Indian first chooses a site in which the concentration in the soil is high. This he determines by sight, touch, and taste, slight differences in these qualities enabling him to distinguish earths containing much saltpeter from other saline earths. The saltpeter occurs only in the surface layer, and this the saltpeter producer, called a *nuniah*, scrapes off with a small flat trowel and mixes with an equal amount of previously extracted earth known as "*bhinjua*" which is described later. This mixture is then carefully packed by treading on a matting of bamboo and straw supported about 4 inches above the ground, that forms the bottom of a filter bed some $4\frac{1}{2}$ feet in diameter and about $1\frac{1}{2}$ feet deep. Water is poured over the mass and allowed to percolate through. The soluble constituents of the soil, including the saltpeter, are carried off in solution and collected in an earthenware vessel. The first portion of this extract which contains most of the saltpeter is boiled down in an open pan over a fire, the fuel for which consists mainly of

dead leaves of bamboos or trees. The ashes from this fire are subsequently used in the preparation of the bhinjua. The boiling of the extract is continued until drops removed from the liquor congeal perceptibly on cooling. The fire is then allowed to die out, and the liquid is ladled, after some cooling, into earthenware pots, where it cools further and deposits a crop of saltpeter crystals contaminated with sodium chloride and other substances. This material is separated from the mother liquor and to it is added the solid residue from the boiling pan, which consists largely of particles of soil and organic matter. This is done in order to convince the salt department that no attempt has been made to separate the salt.

The mother liquor from which the crystals were separated, the later percolations which were considered too weak to boil down, and the ashes from the fire used for the boiling are all added to the extracted earth and the whole mass thoroughly worked to distribute the moisture. It is then spread in the sun to dry, being turned over every 3 or 4 days for a period of about 3 weeks, when the drying is complete. This earth is the bhinjua previously referred to and is stored under cover until the next season. The whole procedure as used by the East Indians appears to be fairly efficient. The mother liquor contains considerable nitrate, and the added ashes contain considerable potash, both of which are conserved by being added to the extracted earth. Chemical changes, due perhaps largely to bacterial action, occur during storage of the bhinjua. Experience has taught that the nitrate content is highest after about 1 year's storage and declines after that. The exact procedure followed doubtless varies considerably with the locality, but the method described may be considered typical.

The nuniah sells the crude saltpeter to a refiner who dissolves it in water and precipitates much of the sodium chloride by boiling off some of the water. The resulting hot saturated solution of potassium nitrate and sodium chloride is allowed to cool, and a fairly pure crop of potassium nitrate crystals separates.

There is some indication that India is considering the possibility of developing its saltpeter industry on a modern basis and reentering the world market. This industry has been described in detail by several writers (13, pp. 772-776; 17; 28; 29; 33; 49). Its chemistry has been discussed by Hamid (23) and Datta (13a).

The older patent literature contains several processes for purifying crude saltpeter. Most of these were no doubt intended to apply to the East Indian product, since this was the main source of saltpeter at that time.

IN OTHER COUNTRIES

The niter industry of inner Mongolia (51) is similar to that of India. About 100 cubic feet of niter-bearing earth are packed on a mat and lixiviated with water. This quantity of earth yields about 64 gallons of percolate which is evaporated to about one-half its bulk and the niter allowed to crystallize out. The dark mother liquor is used to salt out bean curd and as a fertilizer. It is said that each pound of earth yields about 0.2 pound of niter. In crystallizing the niter, two woody stalks of broomcorn are put in either side of the evaporating dish. The niter crystallizes fast around them, and the cake is lifted out, the stalks being used as handles. The saltpeter is recrystallized to yield a good white product.

The industry in Guatemala has been described by Gale (21). Saltpeter is made from the soil in several villages. The process follows the general outline of that used by the East Indians but differs in several respects.

A large part of the saltpeter produced in South American countries is consumed in the manufacture of crude fireworks which are commonly used as an accompaniment to the celebration of festivities and ceremonials of all kinds.

CHILEAN HIGH-POTASH NITRATE

As previously stated, caliche, the crude sodium nitrate ore of Chile, has a potash content ranging from 0.3 to 26.1 percent potassium nitrate. The sodium nitrate runs from 38 to 62 percent. It is not uncommon for the ore at a working, or *oficina*, to average 2 or 3 percent of potassium nitrate, and many operate on ore having 5 or even 7 percent of potassium nitrate. In producing sodium nitrate, this potassium nitrate tends to accumulate in the mother liquor and, although the recovery of a high-grade saltpeter is not particularly feasible, a product known as high-potash nitrate or *nitrapo*, containing from 20 to 80 percent of potassium nitrate with the remainder mostly sodium nitrate, is easily recovered. Such a product was mentioned by Ulex (58) in 1874. Most of that exported to the United States contains from 25 to 40 percent of potassium nitrate. High-potash nitrate or *nitrapo* has been sold to the fertilizer trade in large quantities, particularly in Florida. In the production of a complete fertilizer it is of course advantageous to use this material rather than sodium nitrate, as less potash is required from other sources.

According to Holstein (27) and Faust (16), three methods may be used to produce high-potash nitrate. For those *oficinas* having about 5 percent of potassium nitrate in their ores, the liquor in the crystallizing pans where sodium nitrate is being deposited will yield a high-potash nitrate if transferred to other pans at the proper temperature and the cooling allowed to continue. In the second method the mother liquor from the sodium nitrate crystallization may be evaporated, causing the precipitation of considerable sodium chloride, as this salt is somewhat less soluble in nitrate solutions at high than at low temperatures. High-potash nitrate is deposited when this solution cools. Careful control of the crystallizing operation is required, in order to avoid contamination with magnesium sulphate and boric acid. The third method is based on the fact that the mother liquor from the sodium nitrate precipitation is saturated with potassium nitrate at room temperature and that its solubility decreases more rapidly at lower temperatures than does that of the sodium nitrate. When the liquor cools from 20° to -10° C., about two thirds of the potassium in solution is precipitated to about one third of the sodium nitrate. A mother liquor giving a 25- to 30-percent product by evaporation, without fractional crystallization, will give a 40-percent potash nitrate by refrigeration.

The separation of potassium nitrate from sodium nitrate-potassium nitrate mixtures has attracted the attention of several inventors. Most of these processes appear to be directed at the preparation of saltpeter from crude Chilean nitrate of soda, or from high-potash nitrate. They are mostly leaching and crystallization procedures.

COMPOSTING AND BACTERIAL PROCESSES

Crude saltpeter has been produced by a variety of composting processes in several countries during periods of unusual demand. Thus, France, during the Napoleonic wars, had great difficulty in procuring sufficient niter for the manufacture of gunpowder. The emergency was met by the construction of niter beds or niter plantations. The process consisted in mixing soil, rich in humus or dung, with the debris from buildings, or with lime or wood ashes, and piling the mixture in heaps under sheds to protect it from rain. In some cases a system of gutters was used to distribute urine over the top of the heap. After a time a white film of niter grew on the face of the pile. This was periodically scraped off and leached and the lixivium evaporated and cooled to recover a crop of impure saltpeter crystals. These were purified by recrystallization if necessary. It is said that a similar process is still used in Sweden and some other countries.

Our own Southern States resorted to similar practices during the Civil War. In some areas regular collections of urine were made for use in the production of saltpeter.

One of the earliest patented processes for producing saltpeter is that of Paul Nightingale entitled, "New Method of Making Saltpeter from Vegetables", patented in Great Britain in 1755. His procedure is best described in the language of the patent:

By preparing land and sowing therein the seeds of all kinds of cabbage, savoy, brocoly, borecole, with all the sorts of colewort, Scotch and seacale, purslain, nightshade and all other vegetables of a nitrous quality, which when come to a proper growth are to be gathered and laid on an heap, and to every acre of land sowed with the above vegetables must be added about ten hundredweight of woolen rags, with some kelp or sea weeds, then to be laid in pitts or heaps to ferment and putrifie till they come into earth; the spirit or salt is afterwards to be drawn from the above earth with water and evaporated with fire, as in the manner of making other salts.

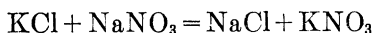
Attempts to utilize the action of nitrifying bacteria have been made by Thorsell and Lundén and by the Société: Aktiebolaget Kväveindustri. The compost processes previously described owe their success at least in part to these bacteria. Hatch and Wilson both propose the production of potassium nitrate from seaweed or kelp. Hatch mixes seaweed with fine sand and a mixture of wood-ash leachings and nitric acid and allows the mass to ferment and develop "blooms", or nodules of nitrate, which are removed and purified to produce saltpeter. Wilson places the kelp in a vat of porous clay and allows it to ferment in the dark until the mass reaches a semifluid condition. The chlorides effloresce on the surface of the vat. The decomposition is allowed to continue until the water has evaporated and the residue is then lixiviated with water to recover saltpeter.

DOUBLE DECOMPOSITION PROCESSES

POTASSIUM CHLORIDE AND SODIUM NITRATE

The most common method of manufacturing potassium nitrate today is by the double decomposition between sodium nitrate and potassium chloride:³

³ Patents of general interest in this connection are those by Bernat, Lambert, Margoles and Aichenbaum, Mercklin and Lösekann, Preussische Bergwerks and Hütten Aktiengesellschaft, Ruer, and Townsend.



This process is commonly known as the "conversion process," and the product is called by many "conversion saltpeter." Anthon (2) in 1858 tells how he undertook, a number of years earlier, an experiment under the direction of an Englishman, W. Thompson, on the preparation of potash saltpeter from Chile saltpeter and potassium chloride. Excellent results were obtained, but no further experiments were made by Anthon until 1851, when he saw an exhibit of potash saltpeter made by this method at an industrial exposition in London. This caused him to resume his researches on the subject which he describes in the reference given. Pick (50) has discussed the procedure in use in Germany in 1875. The modern procedure has been described by Chemnitius (12).

Although the details vary somewhat with the individual plant, the following may be considered the general procedure (46, p. 807): Chile saltpeter is dissolved in about $1\frac{1}{2}$ times its weight of boiling water, and a solution of potassium chloride (usually sylvine from the Stassfurt deposits) in 3 times its weight of water is added to the nitrate solution. Fine granular sodium chloride crystals separate immediately and are removed. The mother liquor is then evaporated to about half its volume, with the separation of another crop of sodium chloride. The sodium chloride is washed with water to recover some of the potassium nitrate adhering to the crystals, the washings being used for dissolving more of the raw material. The mother liquor is further concentrated by evaporation and deposits a crop of crude saltpeter on cooling. This is dissolved in boiling water, and small crystals are produced by cooling this solution while stirring. The crystals, which contain about 0.5 percent of sodium chloride, are dried and sacked for shipment. The success of this procedure is due to the fact that sodium chloride has about the same solubility at high as at low temperatures, whereas that of the potassium nitrate increases sharply with the temperature. Thus, it is possible to eliminate a large part of the sodium chloride at high temperature by boiling or evaporation and to precipitate nearly pure saltpeter when the solution cools. The byproduct sodium chloride from this process is said to be preferred to ordinary salt for pickling meat because of the potassium nitrate it contains. This process is simple and yields a pure product.

This double decomposition has been adapted to the use of crude potash salts such as sylvine or kainite by Lambert and by Nitro-Sel and Margoles. In the processes of Weitz and of Wülfing, the crystallization of the potassium nitrate is rendered more complete by introducing ammonia into the mother liquor after removing the first crop of saltpeter. The remaining ammoniacal liquor is worked up into Solvay soda by introducing carbon dioxide. Pichard has adapted this double decomposition to crude salines low in potash. Nydegger adds potassium chloride to a solution of sodium nitrite and nitrate, and precipitates sodium chloride by evaporation. The mother liquor on cooling yields a crop of potassium nitrate crystals, and the liquor from this step is then treated with enough additional sodium nitrite-nitrate mixture to convert the potassium nitrite present into sodium nitrite and potassium nitrate. Sodium nitrite is crystallized from

this solution, and this mother liquor is again attacked with potassium chloride and the cycle repeated.

POTASSIUM SULPHATE AND SODIUM NITRATE

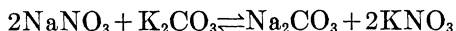
Potassium sulphate may be used in place of the chloride. In the process of J. Michael & Co., a boiling potassium sulphate solution is treated with an excess of sodium nitrate and stirred for about 3 hours at 105° C. The water vapor evolved is replaced, and the precipitated sodium sulphate is separated while hot and washed with water. This sodium sulphate contains some potassium sulphate and is purified by washing with a saturated sodium sulphate solution. On cooling to about 17°, potassium nitrate separates from the mother liquor from which the sodium sulphate has been removed. The final mother liquor is returned to the process.

POTASSIUM CARBONATE AND SODIUM NITRATE

The double decomposition between sodium nitrate and potassium carbonate does not appear to have attracted much attention in recent years. Formerly saltpeter was made from wood ashes or marine-plant ashes by treating the lixivium with sodium nitrate. This process may still be used in some localities. Obviously it fitted in well with the production of potash from wood ashes, a practice that has almost entirely disappeared. In the case of marine-plant ashes a large part of the potash is present as the chloride. Poynter and Patterson prepared saltpeter from kelp or seaweed ash by treating the lixivium of these ashes with sodium nitrate. Potassium nitrate was crystallized out and the resulting liquor processed further to yield bromine and iodine. The process of Theroulde is similar. Osborn says to "lixivate wood ashes with river or rain water at a temperature below 60° F.", which dissolves the "potassa" but not much lime. The lixivium is concentrated to 24° Baumé, transferred to lead vats and cooled. "Upon testing it will now be found that the chloride of sodium has almost ceased to exist as chloride of sodium and becomes chloride of lead which either adheres to the side or is precipitated." The liquor is decanted and if required perfectly chlorine-free—"run a sheet of lead extra through the vat." The decanted liquor is treated with nitric acid previously diluted to 19° Baumé, "with water from the river, or rain water caught at not less than 40 miles from the ocean, to avoid chloride of sodium." The nitric acid is passed with the lixivium through a lead tube or gutter until litmus test shows a slight acid reaction. Potassium nitrate is precipitated out at once and after decanting the mother liquor a second crop may be obtained by evaporating the liquor slightly.

In the process of Richardson nitrate of soda is decomposed by means of a solution of potashes or pearlashes. A crop of saltpeter is obtained and separated; on evaporation a second crop containing both potassium nitrate and sodium carbonate is obtained which is redissolved and the soda rendered caustic with lime. Saltpeter is then crystallized from this solution and the mother liquor boiled down to yield caustic soda. Pennington first converts the pearlash into potassium bicarbonate by passing carbon dioxide from a coke furnace over it, and he then carries out a double decomposition with Chile saltpeter and

recovers potassium nitrate and sodium bicarbonate. The treatment of potash (K_2CO_3) with Chile saltpeter was described by Gentele (22) in 1850, by Wöllner (64) in 1860, and by Schnitzer (54) in 1861. Probably none of these processes is of more than historical interest today. The phase rule chemistry of the reaction



was studied rather extensively by Kremann and Zitek (31) in 1909.

POTASSIUM HYDROXIDE AND SODIUM NITRATE

Both Henry and Newton patented processes in 1858 for the production of potassium nitrate by the reaction of caustic potash with sodium nitrate. This process does not seem to have attracted much attention in recent years.

POTASSIUM SULPHIDE AND MAGNESIUM NITRATE

Potassium sulphide may be decomposed by means of magnesium nitrate. The latter is regenerated with nitric acid and reused. This process is probably an attempt to utilize byproduct sulphide.

POTASSIUM CHLORIDE AND CALCIUM OR MAGNESIUM NITRATE

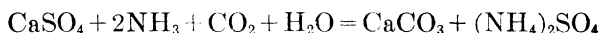
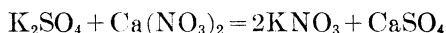
The double decomposition between potassium chloride and calcium or magnesium nitrate has received attention (31a).⁴ Ammonia may be used to effect the separation of the potassium nitrate and calcium chloride that are produced. Lime nitrogen, converted to calcium nitrate with nitric acid or otherwise, has been proposed as a source of the calcium nitrate by the Bayerische Stickstoff-Werke and by Hene and Van Haaren. Hofmann mixes calcium cyanamid with caustic potash or potassium carbonate and a little pulverized copper and basic copper carbonate (to serve as a catalyst) and heats the mass by stirring in a current of air. Most of the nitrogen present is converted into potassium nitrate which is recovered by leaching with water. This process thus provides a direct means of converting lime nitrogen into nitrate without the necessity of first producing nitric acid from the lime nitrogen.

POTASSIUM SULPHATE AND CALCIUM OR MAGNESIUM NITRATE

The insolubility of calcium sulphate has made the double decomposition between potassium sulphate and calcium nitrate attractive. Hampel, and Wolff & Co. and Hampel, in a long series of patents describe several modifications of a cyclic procedure in which, in general, an alkali sulphate reacts with calcium nitrate to give alkali nitrate and calcium sulphate. The sulphate is treated with ammonia and carbon dioxide to give calcium carbonate and ammonium sulphate. Calcium nitrate is regenerated from the carbonate with nitric acid. The raw materials are alkali sulphate, ammonia, and nitric acid. Alkali nitrate and ammonium sulphate are the products. The calcium and carbon dioxide are used cyclically. The cycle

⁴ See processes of E. de Haën Chemische Fabrik, Frowein and Rahlf's, Hampel, I. G. Farbenindustrie, Kubelka and Schneider, Norsk Hydro-Elektrisk KvaestofAktieselskab, Urbain, and Firma Wolff & Co.

involved in Hampel's procedure (24) may be represented by the following reactions:



An important feature of this procedure appears to be the utilization of the sulphate radical of potassium sulphate to produce ammonium sulphate without the expenditure of sulphuric acid. The ammonia is converted into a fertilizer material without first being oxidized to nitric acid. Wolff & Co. and Frowein use a mixture of magnesium and calcium nitrates, made by the reaction of nitric acid with dolomite, for a double decomposition reaction with potassium chloride or sulphate. The calcium nitrate for double decomposition with potassium sulphate is prepared in a process of the Société le Nitrogène by leading nitrogen oxides from the arc process into milk of lime or may be produced in situ by treating calcium carbonate with nitric acid. Henry in a very broad patent describes, among other things, the production of saltpeter by treating sodium nitrate with ammonia and carbon dioxide to give sodium bicarbonate and ammonium nitrate. Lime is added to a solution of the latter to liberate the ammonia and form calcium nitrate solution to which is added potassium sulphate. The lime precipitates as the sulphate, and saltpeter is recovered by evaporation. The I. G. Farbenindustrie finds that the calcium sulphate formed by decomposing calcium nitrate with potassium sulphate is more readily filtered off if the reaction is carried out in the presence of metaphosphoric acid or its salts. Sodium and potassium nitrate are prepared simultaneously in a process of the Chemieverfahren Gesellschaft (French Patent 699,927) by treating artificially prepared glaserite with calcium nitrate and nitric acid.⁵

POTASSIUM CARBONATE OR FLUORIDE AND CALCIUM NITRATE

Bregéat has made the novel suggestion that potassium chloride be fused with silica to give potassium silicate and hydrochloric acid. The former is then treated with carbon dioxide to produce potassium carbonate and silica. Double decomposition of the carbonate is then carried out with calcium nitrate to give potassium nitrate and calcium carbonate.

Calcium nitrate and potassium fluoride have been used by Meyerhofer to produce potassium nitrate and calcium fluoride.

POTASSIUM CHLORIDE AND AMMONIUM NITRATE

Potassium nitrate and ammonium chloride may be prepared by the double decomposition between potassium chloride and ammonium nitrate. In some processes of this type raw potash salts, such as carnallite ($\text{KCl} \cdot \text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), may be used directly as is done by the Kali-Forschungs-Anstalt in German Patent 505,209. In the

⁵ See processes of Kunstdünger-Patent-Verwertungs Aktien Gesellschaft and of Chemieverfahren Gesellschaft.

patents of Osswald and Schad and of the I. G. Farbenindustrie, the ammonium chloride and potassium nitrate are allowed to come down together and then separated by wet elutriation with a liquor of suitable density. Ammonia is added to the alkali chloride—ammonium nitrate solution by the Badische Anilin & Soda-Fabrik to facilitate precipitation of the potassium nitrate. The addition of methyl alcohol also aids this separation (German Patent 406,413). The I. G. Farbenindustrie in British Patents 329,641 and 331,236 carries out this conversion with the aid of liquid ammonia. Potassium chloride is added to Diver's solution (ammonium nitrate in liquid ammonia). Saltpeter crystallizes out, whereas most of the ammonium chloride formed remains in solution.

POTASSIUM SULPHATE AND AMMONIUM NITRATE

Chemische Werke Lothringen and Pfirrmann, and Duns carry out the double decomposition between potassium sulphate and ammonium nitrate in nitric acid solution.

POTASSIUM CHLORIDE AND ALUMINUM NITRATE

The double decomposition between aluminum nitrate and potassium chloride has proved attractive, owing to the fact that the aluminum chloride is readily decomposed to yield hydrochloric acid and aluminum hydroxide, and the latter can be reconverted into nitrate and used again. In a process of the Kali-Forschungs-Anstalt and P. Höfer a solution of aluminum nitrate is stirred with potassium chloride, while heating to about 115° C., and allowed to cool and separate a crop of potassium nitrate crystals. The mother liquor is then evaporated, until nitrous fumes begin to appear. Hydrochloric acid escapes during the evaporation. Sufficient potassium chloride, nitric acid, and water are now added to restore starting conditions, and the cycle is repeated. The aluminum thus serves over and over, and the only raw materials are nitric acid and potassium chloride. In a similar procedure, described by Kaselitz and the Kali-Forschungs-Anstalt, the aluminum chloride is actually removed as the hexahydrate previous to the separation of potassium nitrate. This aluminum chloride is hydrolyzed with steam and reconverted to the nitrate with nitric acid. A mixture of the solid salts,⁶ potassium chloride and aluminum nitrate, may be heated at 200° C. until the evolution of chlorine-bearing gases and nitrous gases ceases and then lixiviated with hot water to dissolve out the potassium nitrate and the unconverted chloride. The nitrate is then separated by crystallization. The aluminum remaining in the lixiviated mass is converted into nitrate, using the nitrous gases, liberated in the first step, which have been freed from chlorine by passing over lime. Sufficient nitric acid or nitrogen oxides to replace that separated as potassium nitrate is added.

Tanaka (57) has studied the solubility relations involved in the double decomposition between potassium chloride and aluminum nitrate. He proposed to obtain the aluminum nitrate by the digestion of clay with nitric acid.

⁶ See Kali-Forschungs-Anstalt British Patent 343,796, and French Patent 693,181.

PROCESSES INVOLVING POTASSIUM COMPOUNDS AND LEAD COMPOUNDS

The older patent literature contains processes for the manufacture of lead compounds, in which potassium nitrate is a byproduct. The main reactions involved in the processes of Cordurié and of Newton are the decomposition of lead sulphate with potassium carbonate in the presence of sodium nitrate to yield potassium nitrate, lead carbonate, and sodium sulphate. Löwe decomposes lead nitrate with potassium carbonate and Stevens decomposes it with the hydroxide. In the process of Delafield saltpeter seems to have been the main product and the lead compounds secondary in importance. Bernhard, in 1865, used lead nitrate and ammonium carbonate to prepare lead carbonate and ammonium nitrate which was then decomposed with lime, liberating the ammonia and obtaining calcium nitrate. A double decomposition of potassium sulphate and calcium nitrate was then carried out to produce potassium nitrate. In more recent years Hampel has described a cyclic process involving lead, in which, however, the lead compounds are not a product but are used over and over again. In this process alkali chlorides are treated with lead nitrate to give alkali nitrate and lead chloride. The latter may be treated with ammonia and carbon dioxide to give the difficultly soluble lead carbonate and byproduct ammonium chloride. The lead carbonate is reconverted to the nitrate with nitric acid and reused in the process, as is also the carbon dioxide liberated in converting the carbonate into nitrate. Instead of converting the lead chloride into carbonate it may be converted into the hydroxide with ammonia or lime and the lead hydroxide reconverted to lead nitrate. The process of Lawarrée is similar. Phosphates play a part in the lead cycle of the Société d'Etudes pour la Fabrication et l'Emploi des Engrais Chimiques. Lead nitrate derived from lead ore by addition of nitric acid yields potassium nitrate on treatment with potassium chloride in a process of the Silesia Verein Chemischer Fabriken.

PROCESSES INVOLVING POTASSIUM COMPOUNDS AND PHOSPHORUS COMPOUNDS

Phosphoric acid or phosphatic materials are used in several processes to prepare potassium nitrate or to produce a fertilizer containing potassium nitrate.

In the processes of Thorssell and Kristensson, of the Chemieverfahren Gesellschaft and of the Kunstdünger-Patent-Verwertungs Aktiengesellschaft crude phosphate may be treated with nitric acid and potassium sulphate to produce calcium sulphate, potassium nitrate, and phosphoric acid. The resulting solution is neutralized with magnesium carbonate, oxide, or hydroxide, which precipitates most of the phosphorus as dimagnesium phosphate. Potassium nitrate is separated by evaporating and cooling the solution.

Holz and Berdell treat phosphate rock with nitric and sulphuric acids. Insoluble calcium sulphate is filtered off, and potassium carbonate or hydroxide is added in order to precipitate dicalcium phosphate which is removed and dried. The solution containing potassium nitrate may be evaporated to dryness and mixed in any desired proportion with the dicalcium phosphate to form a fertilizer.

Dicalcium phosphate and nitrates are also produced by Palazzo and Palazzo by addition of nitric acid to raw phosphate rock and precipitating dicalcium phosphate with potassium or other hydroxide.

The resulting solution contains a mixture of calcium and potassium nitrate. Fractional evaporation yields pure potassium nitrate.

The process of Jost (French Patent 669,492), though not specifying potassium nitrate as a product, could probably be used to produce this salt by substituting potassium chloride for sodium chloride. Phosphate rock is treated with sulphuric acid and the resulting calcium sulphate with ammonia, carbon dioxide, and water, yielding ammonium sulphate and calcium carbonate. To the latter is added nitric acid to form calcium nitrate. The phosphoric acid formed in the first step is converted to alkali phosphate by addition of alkali chloride. Alkali nitrate and calcium phosphate are formed by addition of alkali phosphate to the calcium nitrate.

Monocalcium phosphate and potassium nitrate can be separated from a solution saturated with the two salts at 80° C., according to Griessbach and Röhre, by diluting somewhat and cooling to 30°, whereupon a crop of potassium nitrate separates out. The resulting liquor is evaporated in vacuo at 80°. Monocalcium phosphate separates during the evaporation.

Margoles describes a process for the production of phosphates and potassium nitrate which is carried out in two steps. First, calcium is eliminated from a mixture of calcium nitrate and monosodium phosphate by addition of sodium sulphate to precipitate calcium sulphate. The resulting sodium nitrate-monosodium phosphate mixture is then treated with potassium chloride to form potassium nitrate and monopotassium phosphate, the sodium being eliminated as sodium chloride.

In the process of the Norsk Hydro-Elektrisk Kvaestofaktieselskab potassium chloride is allowed to react with phosphoric acid, tripotassium phosphate and hydrochloric acid are formed. The latter is removed by heating, and the potassium phosphate is treated with hot nitric acid. The resulting solution is cooled until the potassium nitrate crystallizes out.

Jost, in French Patent 669,492 and in British Patents 306,046 and 312,169, converts alkali chlorides with phosphoric acid into phosphates which are then transformed with calcium nitrate into alkali nitrates and calcium phosphate. These may be separated or used together for fertilizer purposes. The tricalcium phosphate may be converted into sulphate and phosphoric acid with sulphuric acid. The calcium sulphate is then converted to carbonate with ammonia and carbon dioxide. The carbonate is converted into nitrate with nitric acid and reused.

PROCESSES INVOLVING INSOLUBLE POTASH MINERALS

In recent years the water-insoluble potash minerals have been much investigated as a source of potash. In several proposed processes potassium nitrate is a direct product. Leucite is especially important in this connection because of the vigorous attempt to establish a potash industry based on the Italian leucites. These processes,⁷ many of which are applicable to other silicates, consist in general in digesting leucite with nitric acid or with oxides of nitrogen, or both (51a), which

⁷ For examples see the following: Norsk Hydro-Elektrisk Kvaestofaktieselskab, British Patent 230,045; French Patents 529,570, 548,064, 579,815, 563,619; and Norwegian Patent 41,581; Jourdan, British Patent 309,957; Canadian Patent 313,910; Danish Patent 41,494; and French Patents 527,065 and 537,039; and Spence & Sons, Ltd., and Craig, British Patent 283,087.

dissolves out potassium, aluminum, and other metallic constituents of the rock. The resulting mixture of nitrates, chiefly those of potassium and aluminum, is treated in various ways to produce potassium nitrate and aluminum nitrate or aluminum oxide. The aluminum oxide is produced by decomposition of the nitrate, the evolved nitrogen oxides being returned to the process. The Chemische Fabrik Rhenania and Messerschmitt in Swedish Patents 35,639 and 35,640, and Messerschmitt in Swiss Patent 94,450 propose to autoclave or boil leucite or other alkali rocks with sodium nitrate or calcium nitrate solution and crystallize potassium nitrate from the resulting liquor.

Processes for obtaining potassium nitrate from silicates in general are numerous. They may be divided roughly into processes in which the rock is digested with nitric acid, as is done by the Norske Aktieselskab for Elektrokemisk Industri in British Patent 125,578, and those in which the rock is digested or roasted with alkalies or salts.

The processes employing digestion with nitric acid vary somewhat but are principally straight acid digestion followed by various means for recovering potassium nitrate from the resulting solution. Berge varies the procedure by first heating the silicate, in this case feldspar, with boric acid to convert the basic constituents into borates which are then treated with dilute nitric acid to form nitrates and free boric acid. Potassium nitrate is recovered from the nitrate solution. Charlton and Shreve suggest an alkaline digestion be applied to New Jersey greensand which consists in autoclaving the greensand with lime, water, and sodium nitrate to form potassium nitrate and sodium hydroxide. These processes enjoy the advantage of an unlimited and cheap source of potassium but suffer from the fact that the silicate always contains other metals that exhaust the nitric acid and the further disadvantages attendant on the disintegration of refractory rocks.

Most proposals for the utilization of alunite (a hydrous sulphate of potassium and aluminum, $K_2O \cdot 3Al_2O_3 \cdot 4SO_4 \cdot 6H_2O$) propose to recover its potassium as the sulphate, since it is originally present in alunite in that form. Detwiller, however, agitates powdered alunite with boiling nitric acid, separates the residual silica by filtration and precipitates the aluminum as the hydroxide by adding caustic potash or ammonia. After filtering off the hydroxide the liquor is agitated with powdered barium carbonate, and the precipitated barium sulphate is removed. Nitric acid is added to the filtrate, and potassium nitrate is obtained by crystallization.

PROCESSES INVOLVING DILUTE OXIDES OF NITROGEN

ABSORPTION IN CARBONATES, BICARBONATES, OR HYDROXIDES

The arc process for fixing atmospheric nitrogen produces a mixture of nitrogen oxides, diluted with a large amount of air. A somewhat similar gas mixture is frequently encountered in the manufacture of nitric acid, due to the difficulty of completely absorbing in water the nitrogen oxides produced in the oxidation of ammonia. One of the favored methods of recovering these dilute oxides, and sometimes more concentrated oxides of whatever origin, is by absorption in alkali or alkaline-earth carbonates, bicarbonates, hydroxides, or oxides. The absorption is sometimes carried out at high temperatures and sometimes at moderate temperatures, using a solution or the

solid material. The resulting product is usually a mixture of nitrates and nitrites, which is then further processed to oxidize the nitrite to nitrate or to separate these elements from each other. A large number of these processes are concerned primarily with the production of calcium nitrate, but they claim or mention the production of potassium nitrate also. This is particularly true of those dealing with the direct oxidation of atmospheric nitrogen.

Brünler and Kettler oxidized atmospheric nitrogen in a special furnace, the mouth of which was submerged in an alkali solution, namely, potassium carbonate; the chloride may also be used. The hot gases passed directly into the solution where the nitrogen oxides formed reacted to form nitrates.

CONVERSION OF NITRITES TO NITRATES

The oxidation of nitrite to nitrate is accomplished by a wide variety of procedures among which are included the direct action of oxygen at ordinary or elevated pressures as in the processes of the I. G. Farbenindustrie (British Patents 293,495 and 306,998) and of Henglein. The treatment of the nitrite-nitrate mixture with nitric acid or oxides of nitrogen has received the most attention. The Norsk Hydro Elektrisk Kvaestofaktieselskab in Norwegian Patent 21,609 describes the separation of nitrates and nitrites by crystallization.

PROCESSES INVOLVING DIRECT ACTION OF NITRIC ACID OR OXIDES OF NITROGEN ON POTASSIUM COMPOUNDS

In the processes discussed so far, except those described under Processes Involving Dilute Oxides of Nitrogen, no attempt is made to utilize the direct action of nitric acid or oxides of nitrogen on potassium salts. As previously stated, the recent development of the synthetic-ammonia industry and the consequent cheapening of nitric acid has aroused much interest in such processes.

POTASSIUM BICARBONATE AND NITRIC ACID OR AMMONIUM NITRATE

Jacobson first converts alkali chloride into bicarbonate by the action of ammonia and carbon dioxide, and then treats the bicarbonate with nitric acid to form alkali nitrate. The carbon dioxide is evolved and used again. Gluud substitutes ammonium nitrate for nitric acid and heats a dry mixture of the former with alkali bicarbonate to volatilize the ammonium bicarbonate formed, leaving alkali nitrate.

POTASSIUM HYDROXIDE OR CARBONATE AND NITRIC ACID

Alkali solutions, such as potassium hydroxide, in a process of the Kali-Industrie Aktiengesellschaft, Thorssell, and Kristensson, may be sprayed into the towers of a nitric-acid plant, and the alkali solution thus neutralized with nitric acid is cooled to separate potassium nitrate after the nitrite has been converted to nitrate. More caustic is added to the mother liquor which is then reused. Classen oxidizes the lower oxides of nitrogen catalytically and absorbs the resulting higher oxides in alkaline solutions to produce nitrates. Potassium nitrate may be produced in this manner.

Ammonia may be oxidized with air or oxygen at temperatures below the decomposition point of the nitrate to be produced, in the presence of strongly basic substances other than magnesium oxide intermixed with metals such as nickel, cobalt, silver, copper, or iron, or their oxides, the latter acting as catalysts. After the gas mixture has passed through the basic substance, the alkali nitrate is leached out with water.⁸ Kassner describes a similar procedure.

POTASSIUM SULPHATE AND NITRIC ACID

Although nitric acid does not ordinarily displace sulphuric acid, it is possible under certain conditions to prepare potassium nitrate by the action of nitric acid on potassium sulphate.

In a process of the I. G. Farbenindustrie Aktiengesellschaft, described in French Patent 666,610, potassium sulphate is treated with hot nitric acid and potassium nitrate crystallized out on cooling. The mother liquor is then saturated with ammonia which brings down ammonium sulphate and part of the remaining alkali sulphate. This mixture may be used as a fertilizer base or treated with fresh nitric acid to obtain more potassium nitrate. Concentration of the mother liquor from the ammonia neutralization causes the deposition of a double ammonium salt of nitric and sulphuric acid known as "leuna salpeter." Another method of the same company⁹ uses 2 moles of nitric acid for 1 mole of potassium sulphate. A good yield of 95-percent potassium nitrate is obtained. Nitric acid is distilled from the mother liquor and reused. Potassium acid sulphate is crystallized from the distillation liquor which is then approximately 70-percent sulphuric acid. Various other methods for carrying out this reaction have been suggested.

Thorsell and the Chemieverfahren Gesellschaft prepare the needed potassium sulphate from the chloride by introducing the latter simultaneously with ammonia into an ammonium chloride solution obtained in the process and containing ammonium sulphate equivalent to the potassium chloride added. The potassium sulphate, which forms as a precipitate, is separated from the mother liquor and stirred with nitric acid and calcium carbonate, the latter originating in the process. Potassium nitrate is formed and remains in solution in contact with the calcium sulphate produced by the reaction. The potassium nitrate may be recovered from this solution by evaporation or by cooling. The calcium sulphate is heated with ammonium carbonate to produce more ammonium sulphate and calcium carbonate.

The sulphate ion may be disposed of by allowing the nitric acid to react on the sulphate in the presence of barium carbonate to produce barium sulphate as a byproduct as in the process of Wolff & Co. and Frowein.

POTASSIUM CHLORIDE AND NITRIC ACID OR OXIDES OF NITROGEN

The direct action of nitric acid on potassium chloride to yield potassium nitrate and hydrochloric acid, $\text{KCl} + \text{HNO}_3 \rightarrow \text{KNO}_3 + \text{HCl}$, is perhaps the most obvious of all processes for saltpeter production. Fraser, in 1857, dissolved potassium chloride in water and treated it with commercial nitric acid. Heat was then applied and the

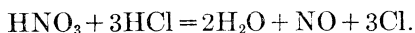
⁸ See processes of Hoffman and of the I. G. Farbenindustrie Aktiengesellschaft.

⁹ See French Patent 669,337, British Patent 310,687, and Norwegian Patent 43,844.

hydrochloric acid driven off and collected. Nearly pure "nitrate of potass" was allowed to crystallize from the liquor. Unfortunately the reaction is not quite so simple as Fraser's invention seems to indicate or as the above equation implies. The difficulties inherent in this procedure have been pointed out by Mehring, Ross, and Merz (45). When potassium chloride is digested with nitric acid, hydrogen chloride is formed and partly escapes from the solution. However, much of this gas remains in the liquor which, as the reaction proceeds, tends toward what may be termed an aqua regia condition, with the resultant liberation of nitrosyl chloride and chlorine, probably by the interaction of the nitric and hydrochloric acids



Considerable nitrogen is lost as nitrosyl chloride unless it is recovered or its formation prevented as suggested by Mirkin (46a). Some nitrogen is probably lost also as oxides of nitrogen by the reaction



That this difficulty is widely recognized is apparent from the patent literature in which are described numerous processes for the treatment of the nitrous gases escaping during the reaction, and processes in which special conditions of temperature, pressure, and concentration are specified for preventing escape of this nitrogen.

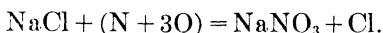
Recognition of the difficulties apparent in the direct treatment of potassium chloride with nitric acid is probably also responsible for the large number of processes already described, in which the nitric acid does not react directly with the potassium chloride but is first converted into aluminum nitrate, lead nitrate, or calcium nitrate and the preparation of the saltpeter then accomplished by double decomposition of the potassium chloride with these compounds. The resulting aluminum, lead, or calcium compounds are reconverted to the nitrate and used again.

If suitable conditions are maintained and the reaction is stopped soon enough, potassium chloride solutions may be treated with nitric acid and a part of the potassium nitrate separated without loss of nitrogen. The resulting mother liquor, however, still contains a large part of the potassium and nitrogen. This mother liquor can be treated in a number of ways, for example, the Kali-Industrie Aktiengesellschaft, Thorsell, and Kristensson, and the Wintershall Aktiengesellschaft, in association with the same inventors, specify that ferrous chloride be added to this liquor to reduce its nitrogen content to nitric oxide which passes off and may be reconverted to nitric acid. After removal of the nitrogen in this manner the hydrochloric acid may be distilled off and collected. The digestion of potassium chloride with nitric acid may be carried out in the presence of other reagents which alter the course of the reaction to some extent. Dow and Telfer, in 1875, added manganese dioxide to the alkali chloride-nitric acid mixture. Chlorine passes off, and the liquor is then evaporated until all the manganese nitrate is decomposed. Alkali nitrate is separated from the mass by lixiviation. A mixture of potassium acid sulphate and potassium chloride may be attacked with nitric acid, according to the inventions of the I. G. Farben-

industrie Aktiengesellschaft described in British Patents 303,351 and 303,355. When the resulting solution is cooled to -5°C. , potassium nitrate of 95 percent purity separates out. The nitrogen remaining in the mother liquor is driven out in the form of nitrogen oxides by treating it with sulphur dioxide at 50° . The nitrogen oxides are washed with water, oxidized to nitric acid, and reused in the process. Hydrochloric acid is recovered from the mother liquor by evaporation in a column apparatus. The residue yields potassium acid sulphate which is also reemployed. The process of Jost is similar.

The I. G. Farbenindustrie Aktiengesellschaft and their assignors, Wild and Beck, in British Patent 283,772, Norwegian Patent 46,993, and United States Patent 1,699,643 claim that better results are obtained if the nitric acid is replaced by a mixture of nitrogen oxides and oxygen used under increased pressures. By the use of suitable concentrations the alkali nitrate separates at once on cooling, without previous evaporation.

Dilute nitric acid¹⁰ is specified by some workers and concentrated acid by others—Dominik, for example. In short, many different conditions and procedures for carrying out the reaction of nitric acid or oxides of nitrogen with alkali chloride solutions have been suggested. J. Skappel passes nitrogen and oxygen along the active surface of a cathode immersed in a fused salt, such as sodium chloride or potassium chloride, and forms nitrates or nitrites directly according to the reaction



Dominik (14) describes a method for the transformation of alkali chlorides into nitrates adapted to the manufacture of nitrogenous fertilizer, by conversion of sylvinite, of low potash content, into nitrate.

A favorite method of treating the gas mixture escaping from the digestion of alkali chlorides with nitric acid is to pass these gases through sulphuric acid where most of the nitrogen is retained and the hydrochloric acid passes on. This is probably best done under increased pressure as described in British Patent 283,771 and French Patent 637,193, both of the I. G. Farbenindustrie Aktiengesellschaft, and United States Patent 1,699,644 issued to Wild and Beck. The sulphuric acid may be denitrated with steam, concentrated, and reused as specified by the Badische Anilin und Soda-Fabrik in German Patent 390,791.

The hydrochloric acid liberated in these processes may be converted to ammonium chloride.¹¹ If this is done at elevated temperatures, the water remains in the vapor phase and solid ammonium chloride is recovered directly. Alkali cyanamid may also be treated with nitric acid to yield alkali nitrate, according to Hene and Haaren in German Patent 311,596.

The action of nitric acid or oxides of nitrogen on solid potassium chloride in various ways has been proposed by several workers. Bronn, Fischer, and Concordia Bergbau, in British Patent 308,028, French Patent 650,030, and United States Patent 1,809,191, treat solid potassium chloride with nitric acid vapor mixed with from 10

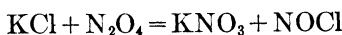
¹⁰ For dilute acid processes see Guye and Darier, British Patent 15,525 (1910); Le Nitrogène Société Anonyme, German Patent 242,014, and Norwegian Patent 20,945; and Guye, Darier, and Van Vloten, United States Patents 1,036,611, and 1,036,833.

¹¹ See Bronn and Concordia Bergbau, British Patent 333,098.

to 15 times its weight of steam. Potassium nitrate is said to be leached out by the condensed steam. Halvorsen absorbs hot dilute nitrous gases in solid compounds, more especially bases, but potassium chloride may be used. Frank, and Siemens & Halske Aktiengesellschaft state that when oxides of nitrogen are passed over halogen salts that the oxides are absorbed and the halogen liberated as such, provided the reaction is carried out in the entire absence of moisture. They do not state at what temperatures the reaction was carried out.

Bergve departs from the usual trend of these processes and blows solid alkali chloride into an electric arc, where it vaporizes and decomposes. The resulting vapors are treated with oxides of nitrogen at such temperatures that nitrates are formed.

Whittaker, Lundstrom, and Merz (62) (see also Ross 52) have found that when nitrogen peroxide is passed over solid potassium chloride at ordinary temperatures in the presence of a small amount of moisture, the reaction



takes place. The nitrogen peroxide is completely absorbed and more than 90 percent of the chloride is readily converted to nitrate, as described in United States Patent 1,965,400 issued to Whittaker and Lundstrom. The nitrosyl chloride may be oxidized to chlorine and nitrogen peroxide and the latter reused in the process. Use of solutions is avoided, and corrosion of the apparatus is thus greatly reduced.

The I. G. Farbenindustrie, in British Patent 310,230 and French Patent 670,561, has suggested that potassium nitrate can be prepared by the action of liquid oxides of nitrogen on potassium chloride. The chloride is digested at 0° C. with liquid oxides containing about 2 percent moisture and then held at the boiling point of nitrogen peroxide for about an hour. The nitrosyl chloride and chlorine formed and the excess nitrogen oxides are separated by fractional distillation.

Several methods of utilizing the nitrosyl chloride from this process or from other processes where it occurs are suggested, as follows: Oxidation to nitrogen peroxide and chlorine, absorption in ferrous chloride with liberation of nitric oxide, reaction with sulphur to give S_2Cl_2 and nitric oxide, reaction with tin to give stannous chloride and nitric oxide, and others.¹² Separation of chlorine from the nitrosyl chloride-chlorine mixture produced in some cases may be accomplished by condensing out the nitrosyl chloride at low temperatures, as is suggested by the Kali-Forschungs Anstalt in French Patent 676,765.

POTASSIUM PENTABORATE AND NITRIC ACID

Hackspill and his associates convert potassium chloride into the pentaborate by treating the chloride with boric acid and driving off the hydrochloric acid formed in the reaction. The borate is then allowed to react with nitric acid to produce potassium nitrate and regenerate the boric acid which is returned to the process.

BYPRODUCT POTASSIUM NITRATE

Potassium nitrate is a byproduct of some industries. It is not always easy to state which product of a given process is the main prod-

¹² See the following patents of the I. G. Farbenindustrie Aktiengesellschaft—British 324,190, French 670,561, and French 680,998; the following of the Kali-Forschungs Anstalt—British 327,047, French 676,765, and German 484,567; and Dominik's United States Patent 1,658,519.

uct and which the byproduct. It is possible that others might consider the potassium nitrate produced in some of the processes already described to be the byproduct. The Chilean high-potash nitrate is, in one sense, a byproduct, but it was discussed earlier in the bulletin because of its importance.

Lürig, as well as Henderson, produced potassium nitrate incidental to the manufacture of soap. Taraud and Truchot absorbed the gases escaping from the Gay-Lussac towers, used in sulphuric acid manufacture, in caustic potash. Potassium salts, including the nitrate, may be recovered in the manufacture of sugar. Boyle, in 1868, obtained potassium nitrate as a byproduct in the manufacture of artificial stone. Sand, gravel, or clay was mixed with soluble potassium silicate and slaked lime and pressed into molds. The mass was then boiled with calcium nitrate, potassium nitrate being formed by double decomposition and recovered from the liquor. The insoluble calcium silicate was left in the mass to act as a binder.

Taylor produces potassium nitrate incidental to the preparation of nitrosyl chloride by passing nitrogen peroxide in contact with potassium chloride solution. The nitrosyl chloride escapes as a gas, and potassium nitrate is recovered from the mother liquor. Sterzl recovers potassium nitrate in a wood decomposition process.

Attempts to recover the potash occurring in the dust carried off from cement kilns by the kiln gases have been numerous in the past. Some of them achieved actual commercial operation. In the processes of Fairbrother and of the Chemical Construction Co., the potash-bearing gases are passed into a sodium nitrate solution where, among other things, potassium nitrate and sodium carbonate are formed by double decomposition. These are separated by crystallization after ridding the solution of hydrosilicates.

POTASSIUM NITRATE FERTILIZERS

A number of processes produce directly a more or less complete fertilizer which contains potassium nitrate or its ions. In the preparation of these fertilizers, potassium nitrate is usually not separated as such but is produced in situ along with other fertilizer ingredients. The discussion of this subject as presented here is comparatively brief, and no attempt is made to list other than a few representative processes. No mention is made of the production of mixed fertilizers by mixing already prepared potassium nitrate with other materials.

A complete fertilizer may be obtained by the processes of the Chemieverfahren Gesellschaft and of Thorssell by treating crude phosphate with potassium sulphate and nitric acid to obtain gypsum and a mixture containing potassium nitrate and phosphoric acid. The latter may be neutralized with ammonia and the whole evaporated to dryness.

A fertilizer containing potash and lime is prepared, according to Schlutius by treating a mixture of crude potassium salts and calcium compounds with nitrogen oxides and steam. In the process of Bosch, a fertilizer containing nitrogen and potash is obtained by simply mixing ammonium nitrate and potassium chloride (both in solid form) in the presence of a small amount of water. A double decomposition takes place slowly in the mixture, so that the final product contains some potassium nitrate and ammonium chloride. This fertilizer is claimed to be nonhygroscopic.

A more involved process is described by Gaus, Griessbach, and Schliephake. Potassium fluosilicate, produced by interaction of potassium chloride with calcium fluoride, silicic acid, and hydrochloric acid, is decomposed with aqueous ammonia to produce silicic acid, which is filtered off, and a solution of potassium fluoride and ammonium fluoride. This solution is treated with another obtained by treating crude phosphate with nitric acid. The fluorine separates as calcium fluoride, which is used again, as is also the silicic acid separated as mentioned above. The solution remaining contains chiefly potassium and ammonium nitrate or phosphate, and when evaporated to dryness yields a mixed fertilizer.

The mother liquor, resulting from the preparation of potassium nitrate by double decomposition of potassium chloride with sodium nitrate, which contains sodium chloride and some potassium nitrate may be further processed to produce a fertilizer.¹³ The mixture of potassium chloride, nitric acid, and hydrochloric acid, resulting from the treatment of potassium chloride with nitric acid to produce potassium nitrate, may be treated with apatite or other tricalcium phosphate to produce a fertilizer containing soluble compounds of nitrogen, lime, phosphoric acid, and potassium. Such a procedure is described by the Aktieselskabet det Norske Kvaelstofkompagni and Halvorsen.

CONCLUSION

It will at once be apparent to those who have read this bulletin that most of the more obvious methods for preparing potassium nitrate have received considerable attention. Present methods and processes are adequate for the production of potassium nitrate for ordinary purposes, but to date no process has produced this material cheaply enough for general fertilizer use. It is doubtful if any process could have produced it cheaply enough for such use prior to the decline in price of nitrate nitrogen that followed the widespread adoption of synthetic-ammonia processes. The present situation is, however, very different, and the interest in potassium nitrate production has increased greatly in recent years, as is evidenced by the large number of recent patents on the subject.

Many of the processes discussed are evidently unsuited to take advantage of cheap nitrate nitrogen, and other processes are offshoots from industries interested primarily in other products. The final solution probably lies in those processes which utilize nitric acid or oxides of nitrogen and potassium chloride.

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PROCÉDÉ DE FABRICATION DU NITRATE DE POTASSIUM. Fr. Pat. 616,286; Oct. 26, 1926.

VERFAHREN ZUR HERSTELLUNG VON KALIUMNITRAT. Ger. Pat. 491,567; Feb. 19, 1930.

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VERFAHREN ZUR REINIGUNG DES KALISALPETERS VON KALIUMPERCHLORAT. Ger. Pat. 81,102; Apr. 22, 1895.

TOWNSEND, J.

IMPROVEMENTS IN THE MANUFACTURE OF NITRATE OF POTASH. Brit. Pat. 2,758; Nov. 6, 1863.

WEITZ, E.

AN IMPROVED PROCESS FOR THE SEPARATION OF ALKALI SALTS. Brit. Pat. 271,869, Aug. 16, 1928; Norw. Pat. 47,061; Oct. 21, 1929.

WÜLFING, J. A. VON

PROCÉDÉ POUR LA SEPARATION DE SELS DES ALCALIS. Fr. Pat. 641,244; Apr. 14, 1928.

POTASSIUM SULPHATE AND SODIUM NITRATE

BRUN and STOERK.

UN MODE DE FABRICATION SIMULTANÉE DU NITRATE DE POTASSE ET DE CARBONATE DE SOUDE PAR LE SULFATE DE POTASSE ET LE NITRATE DE SOUDE. Fr. Pat. 63,123; May 23, 1864.

J. MICHAEL & Co.

VERFAHREN ZUR HERSTELLUNG VON KALIUMNITRAT AUS NATRIUMNITRAT UND KALIUMSULFAT. Ger. Pat. 385,557; Nov. 26, 1923; Fr. Pat. 568,765, Dec. 26, 1923; Brit. Pat. 208,114, June 5, 1924.

POTASSIUM CARBONATE AND SODIUM NITRATE

OSBORN, H. S.

IMPROVEMENT IN THE PREPARATION OF POTASSA. U.S. Pat. 47,562; May 2, 1865.

PENNINGTON, J. C.

IMPROVEMENT IN THE MANUFACTURE OF NITRATE OF POTASH FROM NITRATE OF SODA. U.S. Pat. 35,946; July 22, 1862.

POYNTER, J. E., and PATTERSON, T. L.

IMPROVEMENTS IN OBTAINING OR MANUFACTURING SALTPETRE, IODINE, AND BROMINE. Brit. Pat. 2,112; July 2, 1868.

RICHARDSON, T.

IMPROVEMENTS IN THE MANUFACTURE OF CERTAIN COMPOUNDS OF SODA AND POTASH. Brit. Pat. 2,702; Nov. 29, 1859.

THEROULDE, F. A.

IMPROVEMENTS IN OBTAINING SALTS AND PRODUCTS FROM THE ASHES OF MARINE PLANTS. Brit. Pat. 2,649; Nov. 22, 1858.

POTASSIUM HYDROXIDE AND SODIUM NITRATE

HENRY, M.

IMPROVEMENTS IN THE MANUFACTURE OR PRODUCTION OF SALTPETRE AND THE PREPARATION OF MATERIALS FOR THE PURPOSE. Brit. Pat. 1,819; Aug. 10, 1858.

NEWTON, W. E.

IMPROVEMENTS IN THE MANUFACTURE OF SALTPETRE. Brit. Pat. 1,013; May 6, 1858.

POTASSIUM SULPHIDE AND MAGNESIUM NITRATE

ACTIEN-GESELLSCHAFT FÜR ANILIN-FABRIKATION (Assignors, HEIMANN, H., and ACKERMANN, F.).

VERFAHREN ZUR HERSTELLUNG DER NITRATE DES KALIUMS, BARIUMS UND STRONTIUMS. Ger. Pat. 406,412; Nov. 18, 1924.

VERFAHREN ZUR GEWINNUNG DER NITRATE DES KALIUMS, BARIUMS UND STRONTIUMS. Ger. Pat. 408,116; Jan. 13, 1925.

POTASSIUM CHLORIDE AND CALCIUM OR MAGNESIUM NITRATE

BAYERISCHE STICKSTOFF-WERKE AKT.-GES.

VERFAHREN ZUR HERSTELLUNG VON KALIUMNITRAT. Ger. Pat. 310,661; Sept. 9, 1921.

— and SCHENKE, W.

VERFAHREN ZUR UMWANDLUNG DES KALKSTICKSTOFFS. Ger. Pat. 403,861; Oct. 11, 1924.

FIRMA BAYERISCHE STICKSTOFFWERKE AKT.-GES., and SCHENKE, W.

VERFAHREN ZUR HERSTELLUNG VON ALKALISALPETER. Ger. Pat. 410,924; Mar. 24, 1925.

FROWEIN, F., and RAHLFS, E.

PRODUCTION OF POTASSIUM NITRATE. U.S. Pat. 1,835,704; Dec. 8, 1931. U.S. Patent Office, Off. Gaz. 413: 494. 1931.

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VERFAHREN ZUR DARSTELLUNG VON KALIUMNITRAT UND AMMONIUMCHLORID MIT HILFE VON KALIUMCHLORID. Ger. Pat. 390,394; Feb. 18, 1924.

VERFAHREN ZUR DARSTELLUNG VON KALISALPETER UND CALCIUMCHLORID AUS KALIUMCHLORID UND CALCIUMNITRAT. Ger. Pat. 390,395; Feb. 18, 1924.

HAMPEL, H.

PROCÉDÉ DE FABRICATION D'AZOTATES ALCALINS. Fr. Pat. 551,835; Jan. 15, 1923.

HENE, E., and HAAREN A. VAN

VERFAHREN ZUR UMWANDLUNG VON KALKSTICKSTOFF IN KALISALPETER. Ger. Pat. 332,692; Jan. 21, 1921.

HOFMANN, K. A.

VERFAHREN ZUR DARSTELLUNG VON SALPETER AUS KALKSTICKSTOFF. Ger. Pat. 439,510; Jan. 13, 1927.

I. G. FARBEINDUSTRIE AKTIENGESELLSCHAFT.

PROCÉDÉ DE FABRICATION DE L'AZOTATE DE POTASSE. (CAS A.) Fr. Pat. 681,628; Feb. 3, 1930.

I. G. FARBEAINDUSTRIE AKTIENGESELLSCHAFT. (To JOHNSON, J. Y.)

IMPROVEMENTS IN THE MANUFACTURE AND PRODUCTION OF POTASSIUM NITRATE. Brit. Pat. 328,272; Apr. 14, 1930.

(Inventors, Frowein, F., and Rahlfs, E.)

HERSTELLUNG VON KALISALPETER DURCH UMSETZUNG VON KALKSALPETER MIT KALIUMCHLORID. Ger. Pat. 545,607; Mar. 3, 1932.

KIPPE, O., and KLOCKNER-WERKE AKTIENGESELLSCHAFT.

PROCÉDÉ DE PRÉPARATION DE NITRATE ALCALIN PAR RÉACTION DE CHLORURE ALCALIN AVEC DES NITRATES DE CALCIUM, DE MAGNESIUM, D'ALUMINIUM OU DE FER. Fr. Pat. 748,328; April 10, 1933.

KUBELKA, P., and SCHNEIDER, W.

PROCÉDÉ DE PRÉPARATION DE NITRATE ALCALIN PAR RÉACTION DU NITRATE DE CALCIUM SUR UN CHLORURE ALCALIN EN SOLUTION AQUEUSE. Fr. Pat. 736,815, Sept. 26, 1932; Ger. Pat. 572,722, Mar. 22, 1933.

NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (Inventor, JOHNSEN, H.)

PROCÉDÉ DE FABRICATION DE NITRATE ALCALIN ET DE CHLORURE DE CALCIUM. Fr. Pat. 738,304; Oct. 11, 1932.

[PROCESS FOR PREPARATION OF POTASSIUM NITRATE.] Norw. Pat. 53,372; Dec. 18, 1933. [Original in Norwegian.]

URBAIN, E.

PREPARATION DE NITRATE DE POTASSE. Fr. Pat. 712,335; July 20, 1931.

FIRMA WOLFF & CO., and FROWEIN, F.

VERFAHREN ZUR VERARBEITUNG VON KALIROHSALZEN AUF KALISALPETER.

Ger. Pat. 435,155; Oct. 7, 1926.

FIRMA WOLFF & CO., PULVER- und SPRENGSTOFF-FABRIK (Inventor, HAMPEL, H.)

VERFAHREN ZUR HERSTELLUNG VON KALISALPETER DURCH UMSETZUNG VON MAGNESIASALPETER MIT CHLORKALIUM. Ger. Pat. 413,299, May 5, 1925; Ger. Pat. 413,300, May 2, 1925; Ger. Pat. 414,597, June 4, 1925.

(Inventor, HAMPEL, H.)

VERFAHREN ZUR HERSTELLUNG VON KALISALPETER AUS CHLORKALIUM UND CALCIUM-ODER MAGNESIUMNITRAT. Ger. Pat. 421,988; Nov. 21, 1925.

SHOIKHET, S. N.

[POTASSIUM NITRATE.] Russ. Pat. 31,004; Sept. 30, 1933. [Original in Russian.]

POTASSIUM SULPHATE AND CALCIUM OR MAGNESIUM NITRATE

CHEMIEVERFAHREN GESELLSCHAFT MIT BESCHRÄNKTEN HAFTUNG.

PROCÉDÉ DE FABRICATION DE NITRATE DE POTASSE ET DE CARBONATE DE SOUDE. Fr. Pat. 697,069; Oct. 21, 1930.

PROCÉDÉ DE FABRICATION DE NITRATE DE POTASSE. Fr. Pat. 699,886; Dec. 17, 1930.

PROCÉDÉ DE TRAITEMENT DE SELS BRUTS DE SYLVINITE POUR OBTENIR SOIT DU NITRATE DE POTASSE ET DE SOUDE, SOIT DU NITRATE DE POTASSE ET DE SOUDE ET DU CARBONATE DE SOUDE. Fr. Pat. 699,927; Dec. 17, 1930.

CHEMISCHE FABRIK BUCKAU.

PROCESS FOR THE DECOMPOSITION OF COMPLEX SALTS COMPOSED OF DOUBLE SULPHATES OF POTASSIUM AND CALCIUM. Brit. Pat. 386,854; Jan. 26, 1933.

HAMPEL, H.

VERFAHREN ZUR ÜBERFÜHRUNG LÖSLICHER SALZE IN SALZE ANDERER ART, INSBESONDERE IN KALI- NATRON- ODER MAGNESIASALPETER. Ger. Pat. 321,030, May 17, 1920; Ger. Pat. 335,819, Apr. 22, 1921 (addition to 321,030); Ger. Pat. 337,254, May 23, 1921 (addition to 335,819); Ger. Pat. 345,866, Dec. 16, 1921 (addition to 335,819); Ger. Pat. 365,587, Dec. 4, 1923 (addition to 335,819).

VERFAHREN ZUR HERSTELLUNG VON KALISALPETER AUS KALIROHSALZEN NACH ANSPRUCH 1 BIS 3 DES PATENTS 335,819. Ger. Pat. 374,096, Apr. 19, 1923; Ger. Pat. 380,386; Sept. 4, 1923 (addition to 374,096); Ger. Pat. 381,179, Sept. 17, 1923 (addition to 380,386).

PROCESS OF MAKING NITRATES. U.S. Pat. 1,460,898, July 3, 1923. U.S. Patent Office, Off. Gaz. 312: 159-160, illus. 1923.

HENRY, M.

IMPROVEMENTS IN THE METHOD OF AND APPARATUS FOR TREATING RESIDUA OF DISTILLATION, FERMENTATION, AND SIMILAR OPERATIONS IN ORDER TO OBTAIN AND UTILIZE AMMONIA, CARBONIC ACID, AMMONIACAL SALTS, ALKALINE CARBONATES, SALTPETRE, AND OTHER PRODUCTS. Brit. Pat. 2,483, July 16, 1874.

I. G. FARBENINDUSTRIE AKTIENGESSELLSCHAFT (To JOHNSON, J. Y.)

IMPROVEMENTS IN THE RECOVERY OF PHOSPHORIC ACID FROM CRUDE PHOSPHATES. Brit. Pat. 301,210, Nov. 29, 1928.

— (Inventor, BALZ, O.)

VERFAHREN ZUR GEWINNUNG VON METALLSALZEN DURCH UMSETZUNG VON SULFATEN. Ger. Pat. 514,651, Dec. 15, 1930.

— (Inventors, BALZ, O., and WAGNER, W.)

[METHOD FOR CHANGING CALCIUM SALTS WITH SULPHURIC ACID OR ITS SALTS.]

Swed. Pat. 70,950; Jan. 13, 1931. [Original in Swedish.]

KALI-FORSCHUNGS-ANSTALT G.M.B.H.

HERSTELLUNG VON KALIUMNITRAT DURCH UMSETZUNG VON CALCIUMNITRAT MIT KALIUMSULFAT. Ger. Pat. 536,076, Oct. 19, 1931; Fr. Pat. 731,231, May 24, 1932.

IMPROVED PROCESS FOR THE PRODUCTION OF POTASSIUM NITRATE. Brit. Pat. 377,131; July 21, 1932.

KUNSTDÜNGER-PATENT-VERWERTUNGS- AKTIEN-GESELLSCHAFT (Inventor, LILJEMOTH, F. G.)

PROCÉDÉ PERFECTIONNÉ POUR PRODUIRE DU NITRATE DE POTASSIUM OU UN MÉLANGE DE SELS CONTENANT DU NITRATE DE POTASSIUM. Fr. Pat. 663,113, Mar. 29, 1929.

LE NITROGENE, S. A.

[METHOD FOR SIMULTANEOUS PRODUCTION OF POTASSIUM NITRATE AND SODIUM NITRATE.] Norw. Pat. 19,399, Mar. 23, 1909 [Original in Norwegian]; Fr. Pat. 400,958, June 29, 1909.

NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (Inventor, JOHNSEN, H.)

PROCÉDÉ DE FABRICATION DE NITRATE ALCALIN. Fr. Pat. 738,253, Oct. 11, 1932.

ODDA SMELTEVERK A/S, and JOHNSON, E.

IMPROVEMENTS IN AND RELATING TO THE MANUFACTURE OF FERTILISERS. Brit. Pat. 353,763, July 30, 1931.

SCHWARZENAUER, W.

VERFAHREN ZUR HERSTELLUNG VON KALISALPETER NACH PATENT 321,030. Ger. Pat. 372,407, Mar. 27, 1923.

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PROCÉDÉ DE PRÉPARATION DE PRODUITS DE VALEUR, NOTAMMENT DE NITRATES ALCALINS, À PARTIR DE LA DOLOMIE. Belg. Pat. 378,216, June 30, 1931; Fr. Pat. 732,367, June 14, 1932.

FIRMA WOLFF & Co., and FROWEIN, F.

VERFAHREN ZUR HERSTELLUNG VON KALIUMNITRAT. Austrian Pat. 106,983, Aug. 10, 1927; Ger. Pat. 486,346, Nov. 14, 1929.

WOLFF & Co., PULVER- UND SPRENGSTOFF-FABRIK (Inventor, HAMPEL, H.)

VERFAHREN ZUR HERSTELLUNG VON KALISALPETER AUS KALIHOHSALZEN NACH PATENT 335,819. Ger. Pat. 399,464, July 23, 1924 (addition to 374,096); Ger. Pat. 401,202, Aug. 29, 1924; Ger. Pat. 399,465, July 23, 1924; Ger. Pat. 401,479, Sept. 6, 1924 (addition to 399,465); Ger. Pat. 403,996, Oct. 11, 1924 (addition to 399,465); Ger. Pat. 403,997, Oct. 14, 1924 (addition to 335,819); Ger. Pat. 405,457, Oct. 31, 1924 (addition to 401,479); Ger. Pat. 405,579, Nov. 1, 1924 (addition to 403,996).

— (Inventor, HAMPEL, H.)

VERFAHREN ZUR HERSTELLUNG VON KALISALPETER. Ger. Pat. 399,466, July 23, 1924.

POTASSIUM CARBONATE OR FLUORIDE AND CALCIUM NITRATE

BRÉGEAT, J. H.

AN IMPROVED PROCESS FOR THE MANUFACTURE OF POTASSIUM CARBONATE AND OTHER POTASSIUM SALTS. Brit. Pat. 286,172, Mar. 1, 1928.

MEYERHOFER, A. F.

VERFAHREN ZUR HERSTELLUNG VON KALIUMNITRAT. Swiss Pat. 121,561, July 16, 1927.

POTASSIUM CHLORIDE AND AMMONIUM NITRATE

AICHENBAUM, J.

PROCÉDÉ DE FABRICATION DE NITRATE DE POTASSIUM AVEC COMME SOUS-PRODUITS DU CHLORURE D'AMMONIUM RAFFINÉ ET DU CHLORURE DE SODIUM PROPRE À LA CONSOMMATION OU À TOUT AUTRE USAGE. Fr. Pat. 703,319, Feb. 3, 1931.

BADISCHE ANILIN- & SODA-FABRIK.

VERFAHREN ZUR GEWINNUNG VON KALIUMNITRAT NEBEN KALIUM UND STICKSTOFF ENTHALTENDEN MISCHSALZEN. Ger. Pat. 306,334, Oct. 26, 1920; Ger. Pat. 307,112, Jan. 20, 1921 (addition to 306,334); Ger. Pat. 310,601, Nov. 8, 1920 (addition to 306,334).

(Inventors, MITTASCH, A., and GRIESSBACH, R.)

VERFAHREN ZUR HERSTELLUNG VON ALKALINITRATEN DURCH UMSETZUNG VON ALKALICHLORIDEN MIT NITRATEN. Ger. Pat. 403,844, Oct. 4, 1924.

(Inventors, GRIESSBACH, R., and GIESEN, J.)

VERFAHREN ZUR GEWINNUNG VON KALIUMNITRAT UND CHLORAMMONIUM. Ger. Pat. 406,202, Nov. 14, 1924.

(Inventors, MITTASCH, A., and GRIESSBACH, R.)

VERFAHREN ZUR HERSTELLUNG VON ALKALINITRAT AUS ALKALICHLORID UND AMMONNITRAT. Ger. Pat. 406,294, Nov. 17, 1924.

(Inventor, GRIESSBACH, R.)

VERFAHREN ZUR GEWINNUNG VON ALKALISALPETER UND CHLORAMMONIUM AUS ALKALICHLORID UND AMMONNITRAT. Ger. Pat. 406,413, Nov. 21, 1924.

I. G. FARBENINDUSTRIE AKTIENGESELLSCHAFT (To JOHNSON, J. Y.)

IMPROVEMENTS IN THE MANUFACTURE AND PRODUCTION OF FERTILIZER SALTS. Brit. Pat. 329,641, May 16, 1930.

(To JOHNSON, J. Y.)

IMPROVEMENTS IN THE WORKING UP OF NATURAL AND INDUSTRIAL SALT MIXTURES. Brit. Pat. 331,236, June 25, 1930.

VERFAHREN ZUR GEWINNUNG VON REINEM NATRONSALPETER UND REINEM AMMONIUMCHLORID. Ger. Pat. 476,254, May 13, 1929.

VERFAHREN ZUR HERSTELLUNG VON REINEM KALISALPETER UND REINEM AMMONIUMCHLORID. Ger. Pat. 493,000, Feb. 28, 1930 (addition to 476,254).

[METHOD FOR PRODUCTION OF FIXED AMMONIUM AND ALKALI SALTS.] Norw. Pat. 44,234, July 25, 1927. [Original in Norwegian.]

KALI-FORSCHUNGS-ANSTALT G.M.B.H.

PERFECTIONNEMENTS APPORTÉS AUX PROCÉDÉS POUR L'OBTENTION DE NITRATE DE POTASSIUM EN PARTANT DE CHLORURE DE POTASSIUM ET DE NITRATE D'AMMONIUM. Fr. Pat. 706,300, Mar. 24, 1931.

IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF POTASSIUM NITRATE. Brit. Pat. 360,237, Nov. 5, 1931; Brit. Pat. 360,254, Nov. 5, 1931.

(Inventors, KASELITZ, O. F., HÖFER, P., and ARENS, A.)

VERFAHREN ZUR HERSTELLUNG VON KALIUMNITRAT AUS CHLORKALIUM UND AMMONIUMNITRAT IM KREISPROZESS. Ger. Pat. 505,209, Aug. 19, 1930.

(Inventors, KASELITZ, O. F., HÖFER, P., and POLLATSCHEK, H.)

HERSTELLUNG VON KALIUMNITRAT AUS CHLORKALIUM UND AMMONIUMNITRAT IN KREISPROZESS. Ger. Pat. 536,077, Oct. 19, 1931.

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PROCÉDÉ DE FABRICATION DE NITRATE DE POTASSE ET DE CHLORURE D'AMMONIAQUE PURS. Fr. Pat. 732,225; June 14, 1932.

OSSWALD, P., and SCHAD, K.

PROCESS OF SEPARATING SOLID SALTS OF AMMONIUM AND OF THE ALKALIES OR ALKALINE EARTHS. U.S. Pat. 1,733,272; Oct. 29, 1929. U.S. Patent Office, Off. Gaz. 387: 1081, illus. 1929.

POTASSIUM SULPHATE AND AMMONIUM NITRATE

CHEMISCHE WERKE LOTHRINGEN, and PFIRRMANN, T. W.

VERFAHREN ZUR DARSTELLUNG VON KALISALPETER. Ger. Pat. 353,432; May 18, 1922.

DUNS, H. C. (To the Canadian Industries, Ltd.)

ALKALI METAL SULPHATE CONVERSION. Canad. Pat. 315,880; Oct. 6, 1931. Canad. Patent Off. Rec. 59: 2741, illus. 1931.

POTASSIUM CHLORIDE AND ALUMINUM NITRATE

Höfer, P.

PRODUCING POTASSIUM NITRATE. U.S. Pat. 1,812,531; June 30, 1931.
U.S. Patent Office, Off. Gaz. 407: 1309. 1931.PRODUCTION OF POTASSIUM NITRATE. U.S. Pat. 1,932,587; Oct. 31, 1933.
U.S. Patent Office, Off. Gaz. 435: 1125-1126, illus. 1933.

— and KALI-FORSCHUNGS ANSTALT G.M.B.H.

IMPROVEMENTS IN THE PRODUCTION OF POTASSIUM NITRATE. Brit. Pat. 332,359; July 24, 1930.

KALI-FORSCHUNGS-ANSTALT G.M.B.H.

PROCÉDÉ DE FABRICATION DE NITRATE DE POTASSE. Fr. Pat. 675,201, Oct. 29, 1929; Fr. Pat. 676,714, Nov. 29, 1929; Fr. Pat. 693,181, Aug. 18, 1930; Brit. Pat. 343,796, Feb. 26, 1931.

— (Inventors, KASELITZ, O. F., and Höfer, P.)

HERSTELLUNG VON KALISALPETER AUS ALUMINIUMNITRAT UND CHLORKALIIUM. Ger. Pat. 552,007; Dec. 19, 1933.

— (Inventors, KASELITZ, O. F., and Höfer, P.)

VERFAHREN ZUR HERSTELLUNG VON KALIUMNITRAT DURCH UMSETZUNG VON KALIUMCHLORID MIT NITRATEN. Ger. Pat. 510,092, Aug. 8, 1931; and Ger. Pat. 528,677, Aug. 17, 1931.

— (Inventors, KASELITZ, O. F., and Höfer, P.)

VERFAHREN ZUR HERSTELLUNG VON KALIUMNITRAT AUS ALUMINIUMNITRAT UND CHLORKALIIUM IN KREISPROZESS. Ger. Pat. 533,777, Jan. 24, 1933; Ger. Pat. 538,373, Jan. 24, 1933; Ger. Pat. 554,007, Jan. 24, 1933 (addition to 533,777); Ger. Pat. 555,165, Jan. 24, 1933 (addition to 533,777).

KASELITZ, O.

METHOD OF PRODUCING POTASSIUM NITRATE. U.S. Pat. 1,925,587; Sept. 5, 1933. U.S. Patent Office, Off. Gaz. 434: 156, illus. 1933.

— [F.], and KALI-FORSCHUNGS-ANSTALT G.M.B.H.

AN IMPROVED METHOD OF PRODUCING POTASSIUM NITRATE. Brit. Pat. 327,909; Apr. 17, 1930.

PROCESSES INVOLVING POTASSIUM COMPOUNDS AND LEAD COMPOUNDS

BERNHARD, I.

IMPROVEMENTS IN THE MANUFACTURE OF ARTIFICIAL SALTPETRE. Brit. Pat. 2,142; Aug. 19, 1865.

BERNHARD, and LAGRANGE.

LA FABRICATION SIMULTANÉE D'UN SALPÊTRE ARTIFICIEL ET D'UN BLANC DE PLOMB. Fr. Pat. 67,759; June 19, 1865.

CORDURIÉ.

UN MOYEN DE TRANSFORMER LE NITRATÉ DE SOUDE EN NITRATE DE POTASSE EN PRODUISANT DE LA CÉRUSE. Fr. Pat. 66,926; Apr. 12, 1865.

DELAFIELD, C.

IMPROVEMENT IN THE MANUFACTURE OF SALTPETER. U.S. Pat. 52,145; Jan. 23, 1866.

IMPROVEMENTS IN THE MANUFACTURE OF SALTPETRE AND WHITE LEAD. Brit. Pat. 301, Jan. 31, 1866; Fr. Pat. 70,236, Feb. 6, 1866, addition, May 31, 1866.

HAMPEL, H.

PROCÉDÉ DE TRANSFORMATION DE SELS SOLUBLES EN SELS D'UN AUTRE GENRE, PLUS SPÉCIALEMENT EN AZOTATES DE POTASSE, DE SOUDE OU DE MAGNÉSIE. Fr. Pat. 536,807, Feb. 20, 1922; Norw. Pat. 37,737, Aug. 27, 1923.

PROCÉDÉ DE FABRICATION D'AZOTATES ET D'ACÉTATES. Fr. Pat. 552,364; Jan. 19, 1923.

LAWARRÉE, H.

PROCÉDÉ DE FABRICATION DE NITRATES ALCALINS ET CHLORURE AMMONIÉQUE À PARTIR DE CHLORURES ALCALINS, ACIDE NITRIQUE ET AMMONIAQUE. Belg. Pat. 368,616, Mar. 15, 1930; Belg. Pat. 368,903, Mar. 25, 1930.

PROCÉDÉ DE FABRICATION DE NITRATES ALCALINS. Fr. Pat. 711,363, Jan. 23, 1931; Belg. Pat. 385,838, Feb. 29, 1932; Ger. Pat. 555,166, July 20, 1932; U.S. Pat. 1,891,426, Dec. 20, 1932, U.S. Patent Office, Off. Gaz. 425: 513, illus. 1932.

LÖWE, J. F. F. F.

AN IMPROVED PROCESS OF MANUFACTURING WHITE LEAD. Brit. Pat. 9,122; June 27, 1887.

NEWTON, W. E.

AN IMPROVED PROCESS OF MANUFACTURING SULPHATE OF LEAD, CARBONATE OF LEAD, NITRATE OF POTASH, AND SULPHATE OF SODA. Brit. Pat. 819; Apr. 1, 1859.

SILESIA VEREIN CHEMISCHER FABRIKEN (Inventors, SCHLOSSER, P., BARTSCH, K., and ALASCHEWSKI, G.)

VERFAHREN ZUR GEWINNUNG VON REINEM BLEICHORID UND KALISALPETER DURCH EINWIRKUNG VON SALPETERSÄURE AUS BLEIHALTIGE STOFFE. Ger. Pat. 449,739, Sept. 20, 1927; Ger. Pat. 450,737, Oct. 14, 1927 (addition to 449,739).

SOCIÉTÉ D'ÉTUDES POUR LA FABRICATION ET L'EMPLOI DES ENGRAIS CHIMIQUES (Assignors, JOLIBOIS, P., and CHAUDRON, G.).

PROCÉDÉ DE FABRICATION SIMULTANÉE D'ACIDE PHOSPHORIQUE ET DE NITRATES ALCALINS. Fr. Pat. 691,005; July 1, 1930.

STEVENS, C. A.

IMPROVEMENTS IN THE MANUFACTURE OF WHITE LEAD. Brit. Pat. 16,098; Oct. 31, 1891.

WOLFF & Co., PULVER- UND SPRENGSTOFF-FABRIK (Inventor, HAMPFEL, H.)

VERFAHREN ZUR HERSTELLUNG VON ALKALISALPETER. Ger. Pat. 415,171; June 15, 1925.

PROCESSES INVOLVING POTASSIUM COMPOUNDS AND PHOSPHORUS COMPOUNDS

CHEMIEVERFAHREN GESELLSCHAFT MIT BESCHRÄNKTEN HAFTUNG.

PROCÉDÉ DE FABRICATION DE NITRATE DE POTASSE EN OBTENANT EN MÊME TEMPS DU BIPHOSPHATE DE MAGNÉSIE. Fr. Pat. 682,081, Feb. 10, 1930; Brit. Pat. 329,939, May 29, 1930; Norw. Pat. 47,972, June 2, 1930; Ger. Pat. 539,252, Nov. 26, 1931; Ger. Pat. 558,722, Sept. 10, 1932.

HOLZ, A., and BERDELL, T. VAN D.

IMPROVEMENTS IN THE PRODUCTION OF FERTILIZERS. Brit. Pat. 336,692; Oct. 23, 1930.

I. G. FARBENINDUSTRIE AKT.-GES. (Inventors, GRIESSBACH, R., and RÖHRE, K.)

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